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MANUFACTURE OF CEMENT
MANUFACTURE OF PAPER
MANUFACTURE OF SUGAR
PETROLEUM AND PRODUCTS
MANUFACTURE OF GAS

793

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PREFACE

The International Library of Technology is the outgrowth of a large and increasing demand that has arisen for the Reference Libraries of the International Correspondence Schools on the part of those who are not students of the Schools. As the volumes composing this Library are all printed from the same plates used in printing the Reference Libraries above mentioned, a few words are necessary regarding the scope and purpose of the instruction imparted to the students of—and the class of students taught by—these Schools, in order to afford a clear understanding of their salient and unique features.

The only requirement for admission to any of the courses offered by the International Correspondence Schools is that the applicant shall be able to read the English language and to write it sufficiently well to make his written answers to the questions asked him intelligible. Each course is complete in itself, and no textbooks are required other than those prepared by the Schools for the particular course selected. The students themselves are from every class, trade, and profession and from every country; they are, almost without exception, busily engaged in some vocation, and can spare but little time for study, and that usually outside of their regular working hours. The information desired is such as can be immediately applied in practice, so that the student may be enabled to exchange his present vocation for a more congenial one or to rise to a higher level in the one he now pursues. Furthermore, he wishes to obtain a good working knowledge of the subjects treated in the shortest time and in the most direct manner possible.

In meeting these requirements, we have produced a set of books that in many respects, and particularly in the general plan followed, are absolutely unique. In the majority of subjects treated the knowledge of mathematics required is limited to the simplest principles of arithmetic and mensuration, and in no case is any greater knowledge of mathematics needed than the simplest elementary principles of algebra, geometry, and trigonometry, with a thorough, practical acquaintance with the use of the logarithmic table. To effect this result, derivations of rules and formulas are omitted, but thorough and complete instructions are given regarding how, when, and under what circumstances any particular rule, formula, or process should be applied; and whenever possible one or more examples, such as would be likely to arise in actual practice—together with their solutions—are given to illustrate and explain its application.

In preparing these textbooks, it has been our constant endeavor to view the matter from the student's standpoint, and to try and anticipate everything that would cause him trouble. The utmost pains have been taken to avoid and correct any and all ambiguous expressions—both those due to faulty rhetoric and those due to insufficiency of statement or explanation. As the best way to make a statement, explanation, or description clear is to give a picture or a diagram in connection with it, illustrations have been used almost without limit. The illustrations have in all cases been adapted to the requirements of the text, and projections and sections or outline, partially shaded, or full-shaded perspectives have been used, according to which will best produce the desired results. Half-tones have been used rather sparingly, except in those cases where the general effect is desired rather than the actual details.

It is obvious that books prepared along the lines mentioned must not only be clear and concise beyond anything heretofore attempted, but they must also possess unequaled value for reference purposes. They not only give the maximum of information in a minimum space, but this information is so ingeniously arranged and correlated, and the indexes are so full and complete, that it can at once be made available to the reader. The numerous examples and explanatory remarks, together with the absence of long demonstrations and abstruse mathematical calculations, are of great assistance in helping one to select the proper formula, method, or process and in teaching him how and when it should be used.

Three of the volumes of this library are devoted to subjects pertaining to Applied Chemistry. The present volume contains descriptions of the following industries: manufacture of cement, manufacture of paper, manufacture of sugar, manufacture of gas, and petroleum and products. It has been our endeavor to expound those dominant principles that govern these processes without neglecting those details which are so important in successful manufacturing. The manufacture of cement is carefully treated according to modern practice; the inclusion of a large number of tables and formulas and chapters on the physical and chemical testing of cement will make this treatise of value and interest to the reader. The manufacture of paper and sugar occupy an important place in our industrial life, and the steady progress these industries have made in late years has been taken into careful consideration. Petroleum and Products is perhaps the only attempt to treat on fractional distillation, the refining of petroleum, the manufacture of lubricants and other by-products in a comprehensive, clear, and practical way. The manufacture of coal and water gas, as well as that of acetylene, is fully treated, both practically as well as theoretically.

The method of numbering the pages, cuts, articles, etc. is such that each subject or part, when the subject is divided into two or more parts, is complete in itself; hence, in order to make the index intelligible, it was necessary to give each subject or part a number. This number is placed at the top of each page, on the headline, opposite the page number; and to distinguish it from the page number it is preceded by the printer's section mark (§). Consequently, a reference such as § 48, page 26, will be readily found by looking along the inside edges of the headlines until § 48 is found, and then through § 48 until page 26 is found.

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CONTENTS

MANUFACTURE OF CEMENT	*Section*	*Page*
Introductory	45	1
Relation of Chemistry to Cement Manufacture	45	4
Limestone and the Burning of Lime	45	9
Classification of Limes	45	13
Natural, Roman, and Rosendale Cements	45	17
Manufacture of Natural Cements	45	21
Tests on Natural Cements	45	24
Pozzuolana Cements	45	25
Slag Cements	45	26
Slag Portland Cement	45	29
Silica Portland Cement	45	31
Portland Cement	45	36
Magnesian Cement	45	35
Calculation of Cement Mixtures	45	37
Processes of Manufacture of Portland Cement	45	42
Testing of Portland Cement	46	1
Physical Tests	46	1
Tensile Strength of Various Brands of Cement	46	15
Chemical Analysis of Portland Cement and Raw Materials	46	23
Analysis of Raw Materials	46	27

MANUFACTURE OF CEMENT—*Continued* *Section* *Page*

Notes on Portland Cement 46 32
Location of Cement Works 46 40
Uses of Cement 46 43

MANUFACTURE OF PAPER

Introductory 47 1
Materials Used in the Manufacture of Paper 47 2
Manufacture of Pulp 47 6
Rag Pulp 47 6
Esparto Pulp 47 11
Straw Pulp 47 12
Wood Pulp 47 13
Soda Process 47 15
Recovery of Soda 47 30
Sulphite Process 47 36
Other Processes of Treating Wood . . 47 52
Bleaching the Various Fibers 47 53
Treatment of Bleached Stock 47 65
Beating 47 67
Sizing 47 71
Loading 47 76
Coloring 47 77
Manufacture of Paper from Pulp . . . 47 78
Process for Making Paper by Hand . . 47 78
Process of Making Paper by Machine . . 47 79
Pasteboard or Cardboard 47 91
Parchment Paper 47 92
Water and Its Purification 47 92
Analysis and Tests of Materials Used and of Finished Products 48 1
Apparatus and Chemicals 48 1
Soda Ash 48 11
Causticizing Lime 48 12
Magnesia Lime 48 16
Bleaching Powder 48 19
Bleach Sludge 48 20

MANUFACTURE OF PAPER—*Continued*

	Section	*Page*
Agalite	48	28
Clay	48	30
Ocher	48	32
Ultramarine	48	34
Rosin	48	37
Rosin Size	48	38
Glue or Animal Size	48	40
Salt	48	41
Sulphur	48	43
Bisulphite Liquor	48	45
Caustic Liquor	48	48
Color Value of Pigments	48	49
Analysis of Wood Pulp	48	51
Soda Pulp	48	52
Sulphite Pulp	48	53
Paper Testing	48	54

MANUFACTURE OF SUGAR

Introductory	49	1
Sugar Cane and Its Composition	49	4
Manufacture of Cane Sugar	49	9
Extraction of the Juice	49	9
Fuel and Furnaces	49	13
Defecation and Clarification	49	19
Concentration and Granulation	49	30
Curing the Sugar	49	45
Beet Sugar	50	1
Manufacture of Beet Sugar	50	14
Detailed Description of the Process	50	18
Washing the Beets	50	18
Slicing the Beets	50	22
Diffusion	50	23
Calorization	50	29
Carbonation	50	30
Second Carbonation	50	31
Lime Kiln	50	35
Filter Pressing	50	38

MANUFACTURE OF SUGAR—*Continued*

	Section	*Page*
Sulphuring	50	41
Concentration	50	41
Vacuum Pan	50	45
Osmose Process	50	53
Definitions of the Coefficients	50	55
Stevens Process	50	57
Strontia Process	50	58
Products of Manufacture	50	60
Refining of Sugar	50	64
Sampling Sugars and Molasses	50	70

PETROLEUM AND PRODUCTS

Nature and Composition of Petroleum	51	1
Techno-Chemical Examination	51	8
Natural Gas	51	16
Theories of Origin of Natural Gas	51	19
Production of Crude Oil	51	21
Oil Wells	51	21
Transportation of Crude Oil	51	29
Transportation of Refined Oil	51	32
The Refining of Illuminating Oils	52	1
Chemical Treatment of the Distillate for Burning Oils	52	18
Defining of Naphtha	52	27
The Paraffin Industry	52	33
The Production and Manufacture of Lubricating Oils	52	39
Compounded Oils	52	51
The Manufacture of Vaseline, Waxes, etc.	52	55
Testing Petroleum	53	1
Specific Gravity	53	1
Flashing Point and Fire-Test	53	4
Color Test for Illuminating Oils	53	16
Cloud Test	53	16
Test for Sulphur	53	17
Testing Lubricating Oils	53	20
Viscosity Tests	53	21

PETROLEUM AND PRODUCTS—*Continued*

	Section	Page
Coal Tests	53	24
Railroad Specifications and State Requirements	53	29
U. S. Quartermaster's Specifications	53	31
State Requirements	53	34
Requirements for Bidders	53	35

MANUFACTURE OF GAS

History	54	1
Manufacture of Coal Gas	54	4
Raw Material	54	4
Finished Product	54	6
Process of Treatment	54	8
Distillation of Coal	54	8
Furnaces	54	9
Dampers	54	11
Mouthpieces	54	12
Firing Retorts	54	15
Clogging of Stand Pipes	54	17
Charging the Bench	54	19
Tar Extractor	54	21
Condensers	54	24
Washers	54	25
Scrubbers	54	26
Purifiers	54	27
Gas Meters and Gas Holders	54	32
Ammoniacal Liquor	54	38
Water Gas	55	1
Manufacturing Process	55	3
Water-Gas Apparatus	55	5
Tar Separator	55	11
Oil	55	13
Relief Holder	55	15
Apparatus for the Determination of Carbonic-Acid Gas	55	15
Coke Ovens	55	18
Manufacture of Acetylene	55	23

Manufacture of Gas—*Continued*	*Section*	*Page*
Generators	55	25
Storage Apparatus	55	28
Safety Regulations	55	32
Installing Generators	55	33
Photometry	55	35
Photometers	55	36
Calculation of Candlepower	55	40

MANUFACTURE OF CEMENT

(PART 1)

INTRODUCTORY

1. Historical.—The use of lime as a binding agent for stone, brick, and other materials was known in very early times. How long this preceded the use of natural cement or when the latter was introduced is unknown. It is known, however, that over 4,000 years ago the Egyptians made a natural cement having decided hydraulic properties, and over 2,000 years ago the Romans made a similar cement of excellent quality, which they used for fountains, piers, sewers, water pipes, etc. Prior to this an aqueduct was built at Carthage over 70 miles long, and which at one point crossed a valley where natural cement arches over 100 feet high were used. Some of these are still standing, a testimonial to the high quality of cement manufactured at that date.

In Peru and Mexico, walls have been found so old that the rock has been worn away, leaving the cement mortar projecting from the joints. The time of their construction is only a matter of conjecture.

2. The beginning of the modern practice of cement making is ascribed to John Smeaton, the engineer who constructed the Eddystone Lighthouse in the English Channel. While looking about for a mortar for that purpose he discovered, in 1756, that impure limestones containing a certain amount of argillaceous matter possessed decided

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hydraulic properties when calcined. In his narrative of the Eddystone Lighthouse, when speaking of the Lias deposit in Somersetshire, he says: "This stone when analyzed is found to contain $\frac{3}{16}$ part of its weight of yellowish clay, with a small quantity of sand seemingly of the crystal kind, not quite transparent, but intermixed with red spots." Again, he says, "the fitness of lime for water building seems neither to depend on the hardness of the stone, the thickness of the stratum, nor the bed or matrix in which it is found, nor merely on the quantity of the clay, but on burning and falling down into a powder of buff-colored tinge and in containing a considerable quantity of clay." He further says, "I have seen no lime yet proved to be good for water building but what on examination was found to contain clay; and though I am far from laying this down as an absolute criterion, yet I have never found any limestone containing clay in a considerable quantity but what was good for water building." He also made experiments using lime with trass (a volcanic rock containing pumice), and pozzuolana (a volcanic ash similar to trass consisting chiefly of silicate of alumina), which were previously known to impart hydraulic properties to fat limes. In the Eddystone work he used equal parts by measure of slaked lime and pozzuolana.

In 1786, De Saussure found that the lime of Chamouni possessed hydraulic properties, and he also attributed it to the presence of clay. In 1796, James Parker took out an English patent for "A certain cement or terras (trass) to be used in aquatic and other buildings and stucco work." His process was practically that used in making natural cement at the present time—that is, breaking stone to fragments, burning in a kiln to a point just below vitrifying, and grinding the product. This he afterwards termed "Roman cement." In 1802 natural cement was made from septaria (earthy nodules containing calcite) at Boulogne, and in 1810 Dobbs took out an English patent, which comprised the grinding, mixing, molding into bricks, and drying of raw materials before calcining. This was the beginning of the English slurry process.

3. The first theoretical explanation was offered by Collet-Descotils, professor of chemistry at École de Mines, who stated, in 1813, that the insoluble silica in limestone becomes soluble when heated to a sufficient temperature. He concluded that silica combined with lime during burning, and attributed the hydraulic properties to this.

A more thorough investigation was begun by Vical in 1812 and made public in 1818. His experiments were confined principally to hydraulic lime and pozzuolana, and he showed that all limestone containing a certain quantity of argillaceous matter was capable of producing a hydraulic lime after burning. He explained the changes that take place on burning at a sufficient temperature, and also the causes of setting and hardening. He states that in burning, lime reacts on the clay and combines with it to form a silicate of lime, which is the essential agent of hardening. He further states that silica and alumina form a silicate of alumina that also becomes hydrated and hardens. Alumina, he says, accelerates hardening, but is not necessary to the production of a hydraulic lime. "Iron, manganese, and magnesia are not elements of hydraulicity. Each of these bodies may be lacking without taking away its hydraulic value. Alumina may disappear, but if there is no trace of it, the setting of the lime is less rapid. The essential element of hydraulicity is silica."

In 1818, Canvass White discovered natural-cement rock in New York State, and an industry sprang up there that is still producing large quantities of what is commonly termed *Rosendale cement*. This is similar to Roman cement, and falls under the same class—that is, natural cements. The manufacture of natural cements was begun in New York in 1823, Kentucky in 1829, Maryland in 1836, Illinois in 1838, Kansas in 1868, Wisconsin in 1875; and natural cement is also manufactured in Florida, Georgia, Indiana, West Virginia, Minnesota, Ohio, Pennsylvania, Tennessee, Texas, and Virginia.

In 1824, Joseph Aspdin took out an English patent similar in some respects to Dobbs's, but specifying that definite amounts of clay and limestone be used. The mixing, drying, burning, etc., constitute practically the English process at present. He termed his product *Portland cement*, from the resemblance it bore when hardened to Portland limestone.

4. The first Portland cement made in America was by the Coplay Cement Company, at Coplay, Pennsylvania. David O. Saylor, the president, conceived the idea of making Portland cement from the argillaceous limestone from which they were making natural cement. Early in the seventies, he produced a cement that showed tensile strength equal to the imported, but which after a time disintegrated. Saylor's work was of the experimental and practical order, and realizing the necessity for a study of the chemical composition of his materials, he employed John W. Eckert, a graduate of Lehigh University, and to him is due the credit of demonstrating the adaptability of this new kind of material to Portland-cement manufacture. This marked the beginning of the Portland-cement industry, and from the hundreds of acres of this material in the Lehigh Valley and New Jersey district, thousands of barrels of cement are being made daily. Portland cement is also made in other parts of the United States, but of different materials. Mills are located in New York, Virginia, Ohio, Delaware, Michigan, South Dakota, Arkansas, Kansas, Colorado, California, Tennessee, and Texas; also in Mexico and Canada.

5. Relation of Chemistry to Cement Manufacture. In order to get a thorough understanding of the nature of cement, it is necessary to understand the properties of the elements and materials entering into its composition. This information is given in the preceding Papers on Chemistry. The principal elements met with in cement manufacture, together with their symbols and atomic weights, are given in Table I.

Illustrating, then, the formula $CaCO_3$ is interpreted as follows: 1 atom of calcium, combining, or atomic, weight 39.76; 1 atom of carbon, 11.91; and 3 atoms of oxygen, 3 × 15.88

$= 47.64$. The molecular weight is the sum of the atomic weights—$39.76 + 11.91 + 47.64 = 99.31$. Lime CaO becomes $39.76 + 15.88 = 55.64$. Taking another view of calcium carbonate, we may consider it as a salt composed of lime and carbon dioxide, lime being the base and carbon dioxide the acid. The latter is represented by the formula CO_2, and $CaCO_3$ may be expressed CaO,CO_2. On heating to a sufficiently high temperature, this compound is decomposed, the carbon dioxide passing off as a gas and lime remaining. This is the reaction taking place in a common kiln where builders' lime is burned.

TABLE I

Name	Symbol	Atomic Weight
Aluminum	Al	26.91
Calcium	Ca	39.76
Carbon	C	11.91
Hydrogen	H	1.00
Iron	Fe	55.60
Magnesium	Mg	24.10
Oxygen	O	15.88
Silicon	Si	28.18
Sulphur	S	31.83

Lime will not remain long in the state of CaO, unless kept in a perfectly dry place, as it has a strong affinity for moisture, which it absorbs from the atmosphere, and becomes a hydrate of lime—$CaO + H_2O = Ca(OH)_2$. It also absorbs carbon dioxide and forms a certain amount of calcium carbonate, and, according to Fuchs, a double compound $CaCO_3 + Ca(OH)_2$.

Silicon unites with oxygen, forming silica SiO_2, which occurs in nature as quartz, quartz sand, jasper, opal, etc. It also occurs in numerous compounds known as silicates, and it is in this state that it plays an important part in cement, forming silicates of lime and alumina. A silicate may be

TABLE II

Compound	Formula	Al	Al_2O_3	C	Ca	CaO	CO_2	H	H_2O	Mg	MgO	O	Si	SiO_2
Lime..................	CaO				71.27							28.73		
Slaked lime......... ...	$Ca(OH)_2$				54.08			2.73				43.19		
Slaked lime.............	$Ca(OH)_2$					75.68			24.32					
Calcium carbonate......	$CaCO_3$			11.99	40.03							47.98		
Calcium carbonate......	$CaCO_3$					56.02	43.98							
Magnesia.........	MgO									60.28		39.72		
Magnesium carbonate...	$MgCO_3$						52.21				47.79			
Alumina................	Al_2O_3	53.04										46.96		
Silica..................	SiO_2											52.99	47.01	
Calcium silicate.........	$3CaO,SiO_2$				52.57							34.99	12.44	
Calcium silicate.........	$3CaO,SiO_2$					73.57								26.43
Calcium aluminate......	$2CaO,Al_2O_3$	25.30				37.37						37.33		
Calcium aluminate......	$2CaO,Al_2O_3$		47.69			52.31								

regarded as a salt of silicic acid, just as a carbonate is of carbonic acid, and SiO_2, the anhydride of silicic acid, just as CO_2 is the anhydride of carbonic acid.

Alumina Al_2O_3 is the oxide of aluminum and enters into combination with silica in many complex silicates. Kaolin is an example of a hydrous silicate of alumina whose composition may be given as silica 46.59 per cent., alumina 39.43 per cent., and water 13.98 per cent. Clay is an impure hydrous silicate of alumina and usually contains a larger amount of silica than the above and less water and a small amount of iron oxide and generally small amounts of other constituents, as lime, magnesia, and alkalies, and occasionally manganese or titanium.

6. Composition of Compounds of Importance in Cement.—In Table II are given the percentages of composition of the most important compounds entering into the manufacture of cement.

7. The application of the foregoing may be illustrated by the following example:

Example.—Given a mixture of raw material of the following composition ready for burning:

Calcium carbonate $CaCO_3$	75%
Magnesium carbonate $MgCO_3$	1%
Silica SiO_2	14%
Alumina Al_2O_3	6%
Oxide of iron Fe_2O_3	3%
Alkalies $K_2O \cdot Na_2O$	1%

Assume that calcination is complete and there is no other loss than carbon dioxide and no addition or contamination from fuel used. Calculate the resulting cement.

Solution.—$CaCO_3$ is equal to 56.02 per cent. CaO and 43.98 per cent. CO_2; hence, the loss of CO_2 on calcination, due to the $CaCO_3$, would be

$$75 \times .4398 = 32.98\%.$$

$MgCO_3$ is equal to 47.79 per cent. MgO and 52.21 per cent. CO_2; hence, the loss of CO_2 on calcination, due to the $MgCO_3$, would be

$$1 \times .5221 = .52\%.$$

The total loss on calcination would be

$$32.98 + .52 = 33.50\%.$$

Deducting this loss from 100 per cent., we have

Original mixture	100.00%
Loss of CO_2 on calcining	33.50%
Material available for cement	66.50%

The percentage of CaO in the original raw mixture is

$$75 \times .5602 = 42.02\%.$$

The percentage of MgO in the original raw mixture is

$$1 \times .4779 = .4779\%.$$

Then the theoretical composition of the calcined mixture would be

Lime CaO	$\frac{42.02}{.665}$	$= 63.18\%$
Magnesia MgO	$\frac{.4779}{.665}$	$= .72\%$
Silica SiO_2	$\frac{14}{.665}$	$= 21.06\%$
Alumina Al_2O_3	$\frac{6}{.665}$	$= 9.03\%$
Ferric oxide Fe_2O_3	$\frac{3}{.665}$	$= 4.51\%$
Alkalies $K_2O + Na_2O$	$\frac{1}{.665}$	$= 1.50\%$
Total		100.00%

Theoretically the resulting cement would also have the above composition, but it is never attained in practice, as there is usually from 1 to 3 per cent. of carbonate uncalcined and an addition, very slight, however, of ash from the fuel, which two factors would alter the composition slightly from the above figures. Cement on standing absorbs moisture, which must also be taken into account. The method of mixing raw materials so as to obtain the above results is described under the heading "Portland Cement." Materials of the above composition would produce a poor cement, as seen from chemical analysis, the chief defect being that the combined amounts of oxide of iron and alumina are too high. They produce an easily fusible clinker, but poor cement. Alumina about 7 per cent. and oxide of iron 2 to 3 per cent. would be a better proportion. This is discussed under the heading "Portland Cement."

LIMESTONE AND THE BURNING OF LIME

8. Occurrence and Forms of Limestone. — Limestone is one of the most widely distributed rocks in nature. It is found in almost all countries in stratified beds of considerable extent, showing evidences of deposit (secondary origin) in distinction from igneous or so-called primary rocks. It occurs in all geological formations from the earliest to the present, and some periods and epochs were distinctly limestone-forming times, at least in some sections, while at other points they may have been sandstone or clay-forming periods. The term **limestone** may be broadly used to cover all carbonates of lime, and in this sense may be said to include the perfectly crystallized mineral calcite, the highly crystalline and metamorphic forms known as marble, the microcrystalline form, chalk, the more or less crystalline forms, stalactite, stalagmite, calcareous tufas, etc., deposited from carbonated waters, and lastly, and by far the most widely distributed and extensive deposits of cryptocrystalline and apparently compact form commonly known as blue or gray limestone.

The chemical and physical nature of these varieties differs, depending on the conditions under which they were formed. Calcite, or calc spar, the most perfectly crystallized form showing perfect rhombohedral cleavage, is perhaps the purest and is theoretically the pure calcium carbonate having the chemical formula $CaCO_3$, and, taking the approximate atomic weights that are sufficiently accurate for practical purposes, would have, by weight, the following composition: Calcium, 40 per cent.; carbon, 12 per cent.; and oxygen, 48 per cent. This is the result of ultimate analysis, but gives no idea of the rational formula or grouping of the elements. As calcium is never found native and is only isolated with the greatest difficulty, it is unknown to any save those engaged in scientific pursuits.

Its compound with oxygen, however, lime or quicklime, as it is called, was known in early ages. Grouping the elements therefore as CaO and CO_2, a more comprehensive understanding of its chemical nature may be obtained; that is, 56 parts, by weight, of lime are combined with 44 parts of carbon dioxide to form 100 parts of calcium carbonate. As previously stated, this is its theoretical composition, but the nearest approach to it in nature is in calcite, which very frequently runs above 99 per cent. carbonate of lime and occasionally approaches to within a very small fraction of 100 per cent. The other forms, marble, tufas, etc., contain more or less foreign matter and may run from 99 per cent. down to such a low content of carbonate of lime as to lose their identity as members of the limestone group. As the purer forms are of rare occurrence, they are of no importance in the cement industry, but chalk and limestone running above 80 per cent. carbonate form the foundation.

The principal impurities in limestones are silica, iron, alumina, magnesia, and sulphur. Silica occurs in the free state as sand or combined as silicate of alumina, while alumina occurs only in the latter form. Iron may occur as the carbonate, oxide, or sulphide, and magnesia is generally found as carbonate. Sulphur may occur as sulphide of iron or as sulphate of lime or magnesia. In the burning of lime, any or all of these impurities in small amounts are of very little importance, and if the proper ones occur in larger amounts, the process then passes from the manufacture of lime to the manufacture of hydraulic limes or hydraulic cements.

9. Burning of Lime.—From the foregoing it will be observed that the burning of lime consists in breaking the weakest of the affinities holding together the elements in carbonate of lime—that is, in decomposing it into lime CaO and carbon dioxide CO_2. This is accomplished by means of heat, which process has formed through all ages, and at the present time constitutes an important industry.

The simplest form of burning is in the use of a vertical or shaft kiln. It is circular in section, 8 to 10 feet or more in diameter, and has a vertical height of 15 to 20 feet or more.

Walls may be perfectly straight, forming a vertical cylinder of the above dimensions, or, as is more commonly the case,

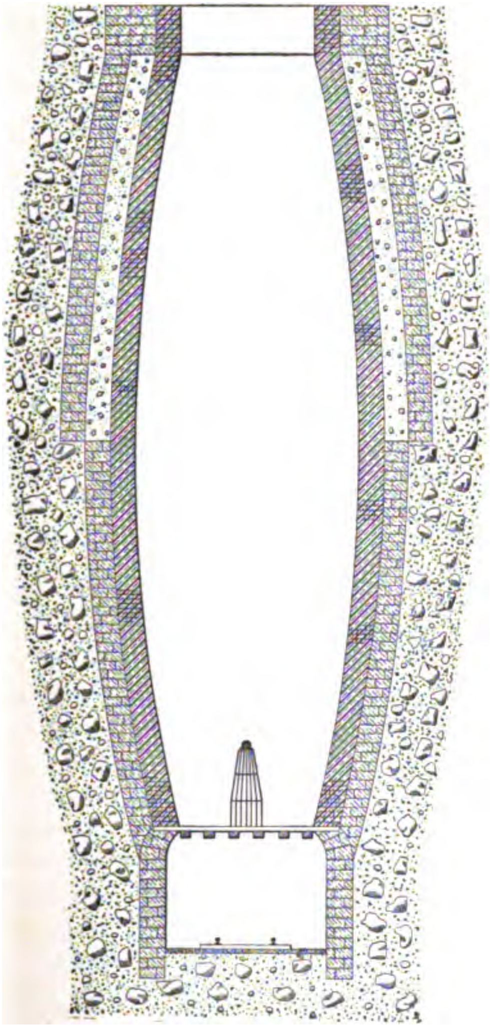


FIG. 1

the walls are bowed, giving a larger cross-section at the center than at the bottom or top. These are the original old forms used for intermittent burning in distinction from the continuous kilns of the present time. In intermittent burning, fuel is charged upon grate bars at the bottom and over this is piled a layer of stone broken to a convenient size. Other layers of fuel and stone then follow until the kiln is full. Fire is then kindled and allowed to burn until all the fuel is consumed, which requires several days or longer, and after the contents of the kiln are sufficiently cool to handle, the burned lime is drawn out below. This is an expensive process, requiring considerable time and expenditure of fuel. A similar style kiln may be operated continuously by drawing the lime from the bottom as burned, the contents above descending, and then adding stone and fuel at the top.

Fig. 1 represents a continuous kiln, but many other forms of both continuous and intermittent types have been designed to meet various conditions. The fuel used may be wood, anthracite or bituminous coal, peat, coke, or natural gas. This industry, although it has suffered considerably from the introduction of natural and Portland cements, is still one of very great importance, and improvements in process of manufacture or handling are being made. The old type of vertical kiln, however, with some modifications, is still universally used.

10. Lime.—The lime as drawn from the kiln consists of hard, rock-like, white, or light-colored blocks, together with more or less fine powder. Lime is highly refractory and resists the highest heat of a furnace without fusing or sintering. Freshly burned lime has a strong affinity for water, and when exposed to moist air absorbs considerable with a marked evolution of heat. This is caused by the chemical union of lime with water, forming calcium hydrate $CaO + H_2O = Ca(OH)_2$, and is known as **slaked lime.** The slaking is accompanied by a considerable increase in volume as well as weight, since 56 parts of lime combine with 18 parts of water to form 74 parts of slaked lime, or calcium hydrate. Slaking may be accomplished either by exposure to moist air or by the addition of water.

11. Importance of Slaking Lime Before Using.—All lime, whether used in sand mortar for masonry or plaster or used almost pure for finishing work in plastering, must be perfectly slaked before using. Very frequently too little attention is paid to this and defective work results. The reason is obvious. If lime is but partially slaked, it consists of a mixture of quicklime CaO and calcium hydrate $Ca(OH)_2$, and when this is mixed with sand and put in work, even with excess of water there may still remain some quicklime. The water evaporates rapidly and the mass may become sound and hard, but it still contains lime having a strong affinity for moisture, and this it must obtain from the atmosphere. The process of hydration may prevent hardening of mortar to a certain extent, but as the amount of free lime grows less hardening advances, and some must become hydrated after the first stage in hardening has taken place. As hydration is accompanied by considerable increase in volume, it causes the mass to swell or expand and peel or crack, thus making very defective work. It is therefore recommended, but not always followed, that lime be thoroughly slaked for at least a week before being used, and in some cases specifications require even a longer period.

12. Classification of Limes.—Limes may be variously classified according to the purity of the rock from which they are obtained, ranging from rock high in calcium carbonate down to those argillaceous forms so low in carbonate that they are no longer considered limestones. As an unbroken series of limes may be formed, varying from the purest to the most impure, no sharply defined limits can be given. The variation in the behavior of these limes in any two successive stages is also so slight as to preclude any possibility of drawing sharp lines between the classes.

13. Rich, or Fat, Limes.—Generally speaking, a **rich,** or **fat, lime** might be said to be one obtained by calcining a stone containing about 95 per cent. or more carbonate. Such a lime slakes readily, with a great increase in volume and accompanied by the generation of considerable heat.

If such a lime be mixed with the exact theoretical amount of water, that is, CaO (40 + 16) 56 parts by weight and water H_2O (16 + 2) 18 parts, a dry powder, calcium hydrate $Ca(OH)_2$ is formed. If an excess of water be used, it works up with an increase of volume into an extremely unctuous paste, being the same thing used by plasterers for the skim or white coat in finishing a wall. In drying, it shrinks considerably, forming a porous mass having practically no tensile strength. The porosity is not nearly so noticeable in the skim coat used by plasterers as when dried in larger masses.

14. Intermediate Limes.—On the same broad basis of classification, an **intermediate lime** may be said to be one produced from a stone ranging from 85 to 95 per cent. carbonate. This lime slakes less readily and is accompanied by less increase in volume and a generation of less heat. A lime of this nature, known to be used largely in this country, is considered by some of its consumers as poor, since they, ignorant of its nature and not observing closely results obtained in the use of it, base their opinion on the long time required to slake it and the lesser amount of heat evolved, while others, equally ignorant of its nature, looking only at results obtained, praise it highly. The fact of the matter is that the impurities, chiefly silica and alumina, put it in a class slightly approaching cement, and in using it there is a weak hydraulic action. A glance at the composition of the stone will show its approach to cement rock, and at the calcined product its approach to cement. The following are approximate analyses, neglecting iron (which acts similarly to alumina), magnesia, and other impurities (each less than 1 per cent.) and assuming that calcination is complete, which is never the case.

Material	Silica. Per Cent.	Alumina. Per Cent.	Lime. Per Cent.	Carbon Dioxide. Per Cent.
Raw stone.....	10	5	47.6	37.4
Calcined stone .	16	8	76.0	

This lime differs from a rich lime in that when hardened after mixing with water it possesses a slight tensile strength. As the stone becomes more impure, it passes into a grade adapted to the manufacture of hydraulic limes.

15. Hydraulic Limes.—These limes are not manufactured to any extent in America, the demand being for natural and Portland cements, the use of the latter growing much more rapidly than the former. In Europe, however, especially France, a considerable amount is used annually. It is made from limestones containing less than 80 per cent. carbonate and, according to tables of Candlot, the limits in chemical composition of these limes are approximately as follows:

Silica	10.0 to 26.0%
Alumina	1.0 to 8.0%
Oxide of iron	.5 to 3.0%
Lime	52.0 to 66.0%
Magnesia	.5 to 4.0%
Sulphuric acid	3.4%
Loss on ignition	8.0 to 21.0%

Hydraulic limes swell less and generate less heat on mixing with water than either of the preceding. They differ also in the property of hardening under water, while the others do not. The length of time required depends on the degree of hydraulicity. In those weakly hydraulic, it varies from 3 to 30 days, while in the more strongly hydraulic, from 1 to 3 days. Those containing large amounts of alumina set more rapidly than those high in silica and low in alumina. In limes, a rise in temperature on addition of water is a normal condition; in hydraulic limes, a rise in temperature, accompanied by rapid hardening or setting, is an indication of poor quality. The specific gravity varies between 2.5 and 2.8, according to the degree of hydraulicity.

16. Calcination of hydraulic limes is accomplished in shaft kilns similar to lime kilns, coal or coke being the fuel commonly used. The rock is broken into pieces about the size of a fist before being charged, in order to facilitate burning and prevent too large an amount of undecomposed carbonate. Small pieces are all the more necessary, since calcination is effected at a low temperature (only a trifle higher than for quicklime) and is not complete, as it will be observed that there is in the finished product a loss of from 8 to 21 per cent. No grinding machinery is used either before or after burning, but a good hydraulic lime should fall to powder when slaked. The slaking is, of course, much slower than in quicklimes, which gradually pass into intermediate, thence hydraulic limes, and finally natural cement. The composition of the raw material may also be such that when calcined at a low temperature it yields a product closely resembling Portland cement calcined and sintered at a higher temperature.

TABLE III

COMPOSITION OF FOREIGN HYDRAULIC LIMES

Locality	Loss on Ignition	Silica	Alumina	Oxide of Iron	Lime	Magnesia	Sulphuric Acid
Germany	5.24	32.60	7.17	6.23	44.96	1.52	1.20
Germany	2.85	34.07	7.49	5.58	46.07	.90	1.96
Germany	2.93	25.21	8.26	8.85	52.46	.50	1.30
Germany	6.78	28.56	4.75	8.24	47.53	2.04	.40
Germany	5.99	25.29	5.41	8.64	50.40	1.24	1.10
Germany		22.14	3.75	3.07	44.22	17.77	
France . .	10.90	17.85	5.20	2.40	56.80	1.35	.75
France . .	12.55	9.75	3.44	1.16	61.85	.60	.45
France . .	9.69	23.13	1.72	.73	63.76	.97	
France . .	8.55	21.60	2.00	1.25	65.80	1.35	.15

PROCESSES OF MANUFACTURE OF CEMENTS

NATURAL, ROMAN, AND ROSENDALE CEMENTS

17. These cements, strictly speaking, all belong to one class, their compositions and the process of their manufacture being similar. They are frequently but erroneously called *hydraulic.* This is a misnomer, since all lime cements, including Portland, are hydraulic. In about 1796, James Parker obtained an English patent for the process of manufacture of a cement that he named *Roman.* He produced it by burning at low temperature a limestone containing about 35 per cent. of clay. This in itself was similar to hydraulic lime, but after burning, instead of slaking with water, it was pulverized, exposed to the air to season for a time, and put on the market in the powdered form. Instead of having a loss on ignition of 8 to 21 per cent., as in hydraulic limes, this loss was less than 5 per cent.

Natural cements, according to Candlot, have a setting time (for a full explanation of this term see *Manufacture of Cement*, Part 2) of from 30 minutes to 8 hours. They contain, in general, less silica and more alumina and frequently more sulphate. In case of Roman and Rosendale cements, both silica and alumina may be either higher or lower than in Portland cement. The name Rosendale in a strict sense is a local one, being applied originally only to cements from the Ulster County district, New York. Other manufacturers in various sections, coming into the market later with similar products, put them on the market as *Rosendale*, whereas natural or Roman would have been a better name. The specific gravity of natural cement is given as 2.7 to 2.9 and Roman as 2.8 to 3.

As there are exceptions to this, it may be neglected, and, as previously stated, all three cements treated as one class. The setting time may range from 5 minutes to as many hours,

TABLE IV

COMPOSITION OF NATURAL CEMENTS FROM VARIOUS COUNTRIES

Locality	Silica	Alumina	Oxide of Iron	Lime	Magnesia	Alkalies	Sulphuric Acid	Carbonic Acid	Water	Loss on Ignition
France, Roquefort	27.20	11.05	4.45	48.05	1.40		1.65			5.35
France, Vassy	24.80	7.00	4.80	44.12	2.08		3.60			7.50
France, Yonne	20.00	8.40	5.70	52.05	.95		2.80			9.60
Russia, Schmidt	14.72	8.56	2.70	36.74	22.26	1.27		10.70		
Russia, Podolsk	12.57	2.76	1.16	48.20	15.10					
Russia, Rosche	17.00	2.07	8.69	45.00	3.81	1.20		19.00	2.01	
England	15.42	5.63	6.81	45.12	.83					25.65
America, Rosendale, N. Y.	27.30	7.14	1.80	35.98	18.00	6.80			2.98	
America, Cumberland, Md.	28.38	11.71	2.29	43.97	2.21	9.00			2.44	
America, Louisville, Ky.	25.28	7.85	1.43	44.65	9.50	4.25			7.40	
America, Milwaukee, Wis.	23.16	6.33	1.71	36.08	20.38	5.27			7.07	
America, Austin, Minn.	18.59	9.14	1.00	40.70	27.00				3.57	

but the former time may be increased by the addition of a little sulphate of lime (raw gypsum or plaster of Paris), as is common practice in the manufacture of Portland cement. The cement on hardening attains a tensile strength greater than hydraulic lime under most conditions, but less than Portland. There are cases on record where hydraulic lime and sand mixtures develop greater tensile strength on long-time tests than natural cements.

The chemical composition varies greatly in different brands, silica, iron, and alumina having wide limits, lime even greater, sulphuric acid still more marked, while the variation in magnesia is exceptionally great. Table IV will show the wide limits in the percentages of various constituents.

A comparison of this table with that of Portland cements (Table IX) will show that for uniformity of chemical composition there is no similarity in cements made in different localities. The extremes seem to show that in some cases silica is less than one-half, that in others alumina is less than one-fifth, oxide of iron less than one-eighth, lime, the chief ingredient, less than seven-tenths, while magnesia has the remarkable ratio of about 30 to 1.

18. All of these cements, however, show decided hydraulic properties, have stood the test of time, and are well suited to work to which they have been applied. In regard to magnesia, which is always less than 4 per cent. in a Portland cement, it should be stated that in a natural cement it possesses hydraulic properties and combines with silica and alumina to form silicates and aluminates, just as lime does, and these compounds when crystallized are stated to be equal in hardness to the lime compounds. The reason for this action in natural cements (when in Portland cements it is said to possess no hydraulic properties and to exercise an injurious effect) may be ascribed to the burning. In natural cements calcined at a low temperature, magnesia hydrates readily, but if the hard and highly burned pieces be picked out and ground, it will be found to hydrate very slowly. In making natural cements high in magnesia, the hard pieces are picked out and discarded. The Portland process differs radically, since all of the material is calcined at a high temperature and brought to incipient fusion.

NATURAL CEMENTS

19. This process differs materially from that of making Portland cement, in that, as a rule, the raw material undergoes no preliminary preparation before burning, but is taken from the quarry or mine direct to the kilns. In several mills there is a sorting process of material into coarse and fine, but these are exceptional. After burning and grinding, the cement is ready for use.

20. Burning the Raw Material.—The kilns are of the vertical type, simple in construction, not unlike a lime kiln, but differing in details. Fig. 2 will serve to illustrate the general type. It consists of a vertical cylinder *A*, 10 feet in diameter and 17 feet high; the bottom part below this is funnel-shaped, tapering to 3 feet in diameter in the next 7 feet, as shown. The kilns are worked on the continuous plan, that is, charging at the top as the material is drawn from the bottom. There is first a layer of stone, then a layer of anthracite, pea, or buckwheat coal, and as the burden descends, successive layers are added. In many cases, no charging is done at night, but the kilns are filled up late in the afternoon and allowed to burn until morning, when the burden is found to have descended some distance. The kiln is then filled and kept full during the

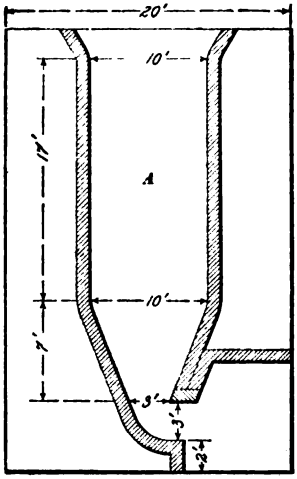


FIG. 2

day. At other places, charging is done by having day and night shifts of workmen to look after it.

As the rock is calcined at a low temperature, the amount of fuel used is small. The material drawn from the bottom is sorted, the hard or overburned pieces being discarded and the remainder put through the grinding mills. Occasionally, when the percentage of overburned material is small (and it is the burner's aim to make it small), the entire product is ground and sent to the stock or packing house. A kiln of the above dimensions produces from 90 to 100 barrels of cement per day.

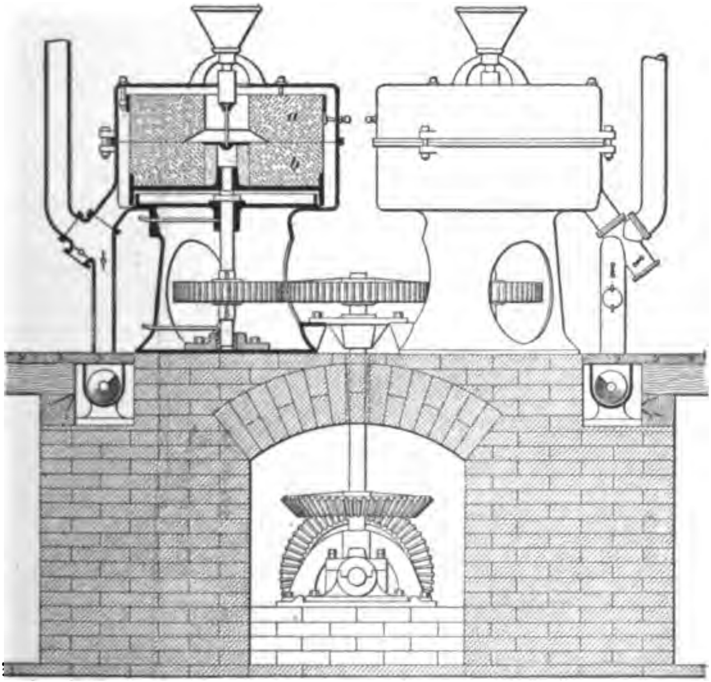


FIG. 3

21. Pulverizing.—The pulverizing machinery differs from that described hereafter in the Portland process in that the ball mills or Griffin mills, which are indispensable in Portland-cement mills, are rarely used. This is due to two reasons: First, the lightly burned rock, corresponding to clinker in the Portland process, is much more readily ground than clinker; and second, the requirements as to fineness, as seen later, are not so rigid as for Portland cement. A common practice in grinding is to put the burned rock through vertical crackers similar to coffee mills, which reduces about 25 per cent. to a merchantable fineness. This is separated by screens and sent to packers. The remainder is pulverized to the required fineness by the old-fashioned mill or burrstones or by rock-emery mills.

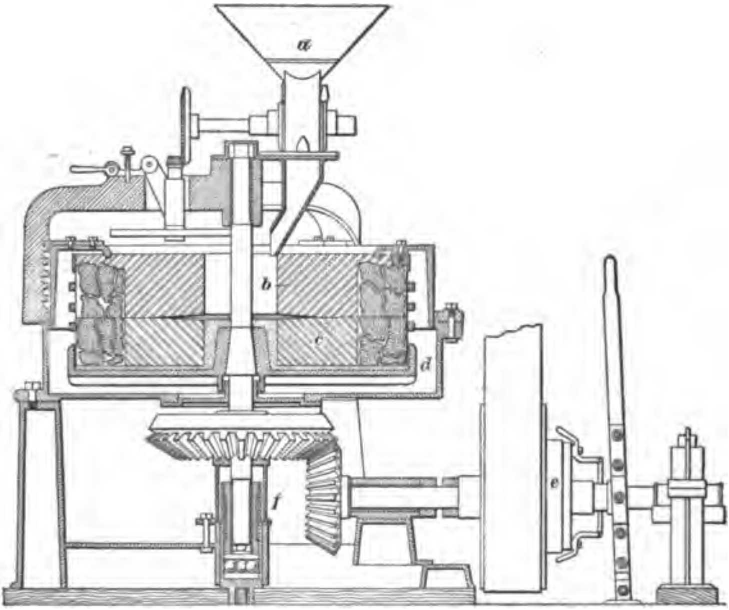


FIG. 4

The former, as will be seen in Fig. 3, consists of two stones *a* and *b* with dressed faces, the lower one *b* revolving and grinding the cement between it and *a*. Each millstone usually consists of one piece of burrstone, millstone, or quartzite. From time to time the stones are redressed, and furrows running radially are cut in to facilitate grinding.

22. Rock-Emery Mill. — This mill is similar to the burrstone, but, as the name indicates, rock emery is used for the grinding surfaces. In Fig. 4 is shown a vertical section of the mill. Material to be ground is fed into the hopper *a*, and falls into the central hole in the upper stone *b*. This stone is stationary, while the lower one *c* is fastened firmly to the bedplate *d*, which is rotated by means of the clutch *e* and gear *f*. Fig. 5 shows the surface of one of the grinding stones. The irregular blocks *a* in the periphery of the wheel are of emery, which is too hard to cut into regular shapes; hence, the blocks are arranged as well as possible and a binder (shown by the white lines) of a metal of special composition is poured around them, the whole being bound together by shrinking an iron band around the circumference. The triangular segments *c* and the bands *d* radiating from the center are made of a poorer grade of emery or burrstone, which, being softer, wears more readily and permits the peripheries of the wheels to come in closer contact.

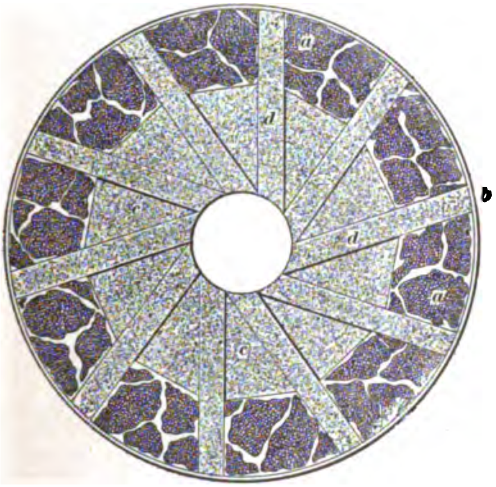


FIG. 5

As will be readily seen, the material to be ground must undergo a preliminary crushing, as the mill is only adapted to fine pulverizing.

TESTS ON NATURAL CEMENTS

23. *Fineness.*—The fineness required by specifications is not such as for Portland, it being coarser. It is determined as described in *Manufacture of Cement*, Part 2. The screens recommended are 50 meshes to the linear inch, Stubb's wire gauge, No. 35; 74 meshes to inch, Stubb's, No. 37; and 100 meshes, Stubb's, No. 40. A natural cement of which 98 per cent. passes 50 mesh, 85 per cent. passes 100 mesh, and 30 per cent. passes a 200 mesh is considered sufficiently fine.

Setting Time.—The time of setting should be from 10 to 30 minutes (see "Portland Cement").

Tensile Strength.—(For explanation see "Portland Cement.")

The following strengths were recommended and given as an average of natural cements by the committee of The American Society of Civil Engineers, 1899, appointed for that purpose, although frequently much higher results are obtained.

TENSILE STRENGTHS OF NATURAL-CEMENT MIXTURES

NEAT (*Cement and Water*)

	Pounds
One day—until set in air and for the balance of the 24 hours in water	40 to 80
Seven days—1 day in air and rest of time in water	60 to 100
Twenty-eight days—1 day in air and rest of time in water	100 to 150
One year—1 day in air and rest of time in water	300 to 400

SAND, 3 PARTS; NATURAL CEMENT, 1 PART

Seven days	30 to 50
Twenty-eight days	50 to 80
One year	200 to 300

POZZUOLANA CEMENTS

24. Pozzuolana, or **puzzolan cements,** are obtained by mixing powdered hydrate of lime with natural or artificial materials without subsequent burning. These materials, as a rule, have no hydraulic properties in themselves, but become hydraulic when united with lime and ground to extreme fineness. The natural materials used are volcanic products, such as pozzuolana, trass, and santorin earth, and the artificial materials used are furnace slag, burned clay, etc. The mixture with pozzuolana, which is found in great quantities around Naples, was known to the Romans, who used it in all their important works, and it is still used in Europe. The percentages of the most important constituents of several of these materials are given in Table V.

TABLE V

Material	Silica	Alumina	Oxide of Iron	Lime	Magnesia	Soda	Potash
Pozzuolana	38.50	18.35	14.90	8.70			
Pozzuolana	42.00	15.50	12.50	9.47	4.40		
Pozzuolana	59.10	21.20	4.76	4.70			
Pozzuolana	53.20	8.75	21.00	10.30			
Trass	48.93	18.95	12.34	5.40	2.31		
Trass	57.50	10.10	3.90	7.70	7.10		
Trass	46.60	20.60	12.00	3.00			
Santorin earth	66.37	13.72	4.81	2.98	1.29	4.22	2.83

In using the above materials, the proportions have been various, and in some places in Europe excellent results have been obtained. These cements were never used to any extent in the United States, as the amount imported, while increasing, is still comparatively small.

25. The following mixtures, used in several European countries, are recommended.

FRANCE

	Parts
Trass	3.0 or 3
Hydraulic lime	5.0 or 6
Sand	6.5 or 5

BELGIUM

Trass	2
Hydraulic lime	6
Sand	4

GERMANY

Trass	2 or 3
Hydraulic lime	3 or 3
Sand	4 or 3

ENGLAND

Pozzuolana	1
Hydraulic lime	1

SLAG CEMENTS

26. Adaptability of Slags.—In recent years, a process has been developed for producing a cement from blast-furnace slag. Plants for this purpose have been built in Germany, England, and Belgium and there are several in operation in the United States. In blast-furnace practice, limestone is used to flux off silicious matter, which yields a slag high in lime. It has long been held that this material could be used in the manufacture of cement and the truth of the same has been demonstrated. As the iron ores used vary in composition, it follows that the charges, and consequently the slags, vary, and it is not all slags that are adapted to the purpose. There are, however, a number of ores that yield slags within limits adapted to their use in the manufacture of cement. The following analysis shows the composition of such slags:

Silica	30.0 to 35.0%
Lime	45.0 to 50.0%
Alumina	10.0 to 15.0%
Ferrous oxide	.2 to 1.5%
Magnesia	.5 to 3.5%
Sulphuric acid	.2 to .0%
Manganese	3.0 to 4.0%

The above show considerable lime in combination with silica and alumina, but not sufficient to form a cement, and the problem has been to add sufficient lime to bring its content high enough and the silica and alumina low enough.

27. Preliminary Treatment and Manufacture.—As the cold slag is hard and glossy, rendering it extremely hard to pulverize, it undergoes a preliminary treatment as it comes from the furnace. While still hot and fluid it is run into an inclined cast-iron trough through which a strong current of water flows. This has the effect of granulating it and making it brittle, thus preparing it for the necessary pulverizing to follow. It also effects a desirable chemical change—namely, the decomposition of the calcium sulphide, which is injurious to cement. The calcium combines with the oxygen of the water and forms lime CaO and the sulphur forms hydrogen sulphide and escapes as gas.

$$CaS + H_2O = CaO + H_2S$$

The wet slag sand is then passed through a drier somewhat similar to those described—or of any other suitable design—and, being thoroughly dried, is ready for mixing. It is then added to about an equal amount of crushed limestone of proper quality and 3 to 4 per cent. of slaked lime, and the entire mass is finely ground either by processes heretofore described or other suitable machinery. Success here is dependent on a very intimate mixture of the raw materials. It is necessary that the fineness should be such that not more than 5 per cent. is left on a 100-mesh sieve and 15 to 20 per cent. on a 200-mesh sieve. This mixture of slag sand, limestone, and hydrate of lime is mixed with

8 to 10 per cent. of water and molded into bricks of 7 or 8 pounds. These are stored for a few days, when, owing to the hydrate of lime, they become resistant enough to permit handling and charging in the kiln. This is designed especially for the purpose and is of the shaft-kiln type, but differs in that compressed air is admitted through a double row of tuyeres at different points. This is to burn any carbon monoxide or carbureted hydrogen formed, as well as to concentrate heat at the point where clinkering is desired. The clinker is drawn from below and sprinkled with a little water, to convert any quicklime into hydrate of lime, after which it is stored under cover for a few weeks before the final pulverizing, which is done in ball and tube mills described later.

28. In chemical composition, slag cements run lower in lime and higher in silica than Portland cement, as will be seen from the following average analyses (Table VI).

TABLE VI

COMPARATIVE COMPOSITION OF SLAG AND PORTLAND CEMENTS

Determinations Made	Portland Cement. Per Cent.	Slag Cement. Per Cent.
Loss on ignition	1.00	3.69
Silica	21.50	24.93
Alumina	8.00	11.61
Oxide of iron	2.50	2.30
Lime	63.00	51.59
Magnesia	2.50	4.46
Undetermined	1.50	1.42
Total	100.00	100.00

Physical tests up to one year show lower results than Portland cements and their value as indicated by long-time tests has not been demonstrated. The success of slag cement as made by the foregoing process is not comparable with Portland cement, and in Europe the process is being abandoned, but in America, where a different method is used, there is no means of predicting what the outcome may be.

At present, slag cement and slag Portland, described in the following pages, are rejected in specifications calling for Portland cement. As to whether they should be considered true Portlands or not is open to discussion and largely a matter of opinion, as there are good arguments pro and con. A few of the differences will be summed up for comparison.

Portland cement contains from 59 to 66 per cent. lime.

Slag cement contains from 40 to 59 per cent. lime.

Portland cement has a specific gravity of 3.00 to 3.20.

Slag cement has a specific gravity of 2.70 to 2.90.

Portland cement sets in from 1 to 8 hours.

Slag cement sets in from 8 to 10 hours or longer.

Portland cement has less than 2 per cent. sulphuric acid.

Slag cement may have as high as 3 or 4 per cent. sulphuric acid.

This last statement is true only of European slag cements, as in American brands the sulphur is very much lower.

NOTE.—There are no particular reasons for calling the cement just described *slag cement* and the one following *slag Portland cement*, as they are similar in properties and should both be called *slag cements* or *slag Portlands*. The confusion in names arises from the fact that the process just described is a Belgian process and the product is known as slag cement, while American manufacturers gave the name slag Portland to the rotary-kiln slag cement. The confusion here is similar to that in the Roman, Rosendale, and natural cements.

SLAG PORTLAND CEMENT

29. Process of Manufacture.—Slag Portland is a name given to a cement intermediate in the materials and process of manufacture between slag cement and true Portland cement. In raw materials used, it resembles slag cement;

in process of manufacture, rotary-kiln Portland cement. It is made mainly of slag from blast furnaces, using an ore requiring such a flux that it produces a slag of suitable composition to be mixed with high-grade limestone. As slag takes the part of clay (see discussion under heading "Portland Cement"), it will be readily seen that all slags are not suitable for this purpose any more than are all clays. The slag is run hot from the furnace into a stream or tank of water, which granulates it to about the size of a pea or smaller. In this state it is usually light in color, porous, and low in specific gravity. The slag and limestone are carefully analyzed and a proper cement mixture made up, either before or after the first grinding, according to the method hereafter described. The rest of the process, grinding and burning the mixture and grinding the clinker, is identical with the Portland process.

30. Properties.—Slag cement is light in color; slag Portland is dark in color (it should be noted that the color is due to chemical composition more than to the process of manufacture). It is usually finer than Portland and not gritty. It works up into an unctuous paste when mixed with water and stands boiling tests. Pats exposed to the

TABLE VII

ANALYSES OF SLAG PORTLAND CEMENTS.

Sample	Silica	Alumina	Oxide of Iron	Lime	Magnesia	Sulphuric Acid	Loss on Ignition	Carbon Dioxide
1	32.00	3.26	1.34	54.42	1.73	.61	6.40	
2	26.98	12.67	.49	51.02	2.40	None	4.60	
3	26.60	9.29	3.11	50.18	3.82	.68	4.10	
4	28.20	13.93		33.20	16.56	.11		
5	28.60	12.29	.51	50.53	1.44	.07	4.70	
6	22.20	7.76	4.34	64.25	.58	.41		
7	27.20	14.18		50.33	3.22	.15		4.25

air must be covered, to prevent cracking. It discolors brown or yellow when set in air, but if allowed to harden in water does not show this. Briquettes hardened in water show a green color on a fresh fracture, but on exposure to air turn white, due to the oxidation of calcium sulphide or iron sulphide. All slag cements do not show this, but those that do should be used only for the interior of walls or underground work. It may be used to adulterate Portland, or ground slag may be introduced into Portland. To detect such adulteration, a specific-gravity test is sufficient, as it is not as heavy as Portland cement, a cubic foot weighing about 70 pounds, while Portland weighs from 80 to 90 pounds. Its chemical composition may be seen from Table VII, each analysis representing a different brand.

SILICA PORTLAND CEMENT

31. Process of Manufacture.—The process for making this cement was first introduced in Denmark under the claim of the special advantage of making a mortar impermeable to moisture and able to resist the destructive influence of the elements. It is made by pulverizing to extreme fineness equal parts of Portland cement and clean, dry silica sand. This is effected by weighing out equal portions and passing through a tube mill, as described under the heading "Portland Cement."

32. Properties.—The resulting mixture is slow setting, is slightly lower in specific gravity than Portland, which, in connection with its extreme fineness, makes it weigh less, bulk for bulk. Owing to the difficulty in always filling a measure under exactly the same conditions, that the degree to which it is packed may be the same, the old method of judging by weight per bushel should be discarded. Specific-gravity tests, likewise, do not find much application in practical work, as a variation in density is caused by a variation in the burning of the Portland. The only methods of comparison, then, of silica Portland and Portland cements are by observations in actual work or by tensile-strength tests.

In Portland cement, tests are made on strength of pure cement gauged with water (such a mixture is called *neat mixed* or *neat*) and cement and sand mixed with water. In samples of the same cement ground to different degrees of fineness, it is observed that when worked up neat the coarser samples develop a higher strength and the finer a lower strength; but when mixed with sand in various proportions, the finer samples show higher results than the coarser samples. In explanation of this, it may be stated that in the finer cements worked neat, while the energy of crystallization is greater, the strength is dependent entirely on the adhesive and cohesive power of the crystals upon one another. In the coarser samples, mixed neat, the coarse particles act as inert matter chemically, but as sand physically, and the finer particles in crystallizing interlock and exert an adhesive force upon these coarse particles that is greater than the adhesive power among the fine particles, making the combined adhesive and cohesive powers of the coarse cement exceed those of the fine cement. In sand mixtures, it is found that the finer cement develops higher strengths, owing to the sand furnishing the particles for cement for adhesive purposes and the fine grinding of cement making the chemical energy of crystallization greater. In other words, in a cement or cement mixture, the coarse particles, whether they be unground cement or sand, act as so much inert matter chemically, but form a bond physically for the adhesion of the finer particles in crystallizing. It seems rather inconsistent to state that a mixture of 1 part Portland cement and 1 part sand, to which sand equal to three times their combined weight has been added, should give as high tests under certain conditions as a mixture of 1 part of pure cement and 3 of sand, the first mixture being 7 parts sand and 1 part cement, the second but 3 parts of sand and 1 part of cement, but such is claimed to be the case by silica-cement supporters.

33. The following tensile strength tests on a Portland cement and a silica Portland made from the same will serve to illustrate:

PORTLAND CEMENT

	Breaking Strength in Pounds
Neat (cement and water):	
One day in air, 6 days in water	500
One day in air, 27 days in water	600
Sand, 3 parts; cement, 1 part:	
One day in air, 27 days in water	200

SILICA PORTLAND CEMENT

	Breaking Strength in Pounds
Neat:	
One day in air, 6 days in water	350
One day in air, 27 days in water	450
Sand, 3 parts; cement, 1 part:	
One day in air, 27 days in water	200

In the neat tests, it will be observed that the Portland cement shows up higher than the silica Portland, which may be explained by the fact that the latter contains 50 per cent. of sand that is inert not only chemically, but also physically, to a great extent, since it is ground to an exceeding fineness, while the former contains no silica, as sand, but being coarser, contains particles of cement that are inert chemically, but act as sand physically. It should be added that the fineness of these two cements was as follows:

Kind of Cement	Passing a 100-Mesh Sieve. Per Cent.	Passing a 200-Mesh Sieve. Per Cent.
Portland	92	65
Silica Portland	100	93

The seeming inconsistency heretofore mentioned may be further explained by stating that the energy of chemical action is greater the more nearly we approach the molecule in fineness, or more properly speaking, the more nearly we approach the molecule, the larger is the per cent. of the cement that reacts chemically and the lower the per cent. of that acting as a binding agent only. This explains results obtained in both neat and sand tests.

The composition of the preceding cements and the tests thereon are taken from a paper by M. T. Butler, the analyses being as given in Table VIII.

TABLE VIII

COMPARATIVE ANALYSES OF PORTLAND AND SILICA PORTLAND CEMENTS

Constituents	Portland Cement. Per Cent.	Silica Portland Cement. Per Cent.
Silica	22	61
Alumina and oxide of iron	12	6
Lime	62	31
Magnesia, sulphuric acid, etc.	4	2
Total	100	100

34. The manufacture of silica Portland cement has been carried on quite extensively in Canada during the past few years, and several plants are operated in the United States. The sales, however, up to 1902 are not to be compared to those of Portland cement. The merits of the case will have to be demonstrated more fully and more broadcast, and the same objections and prejudices that met American Portland at its introduction overcome. Should it be shown that for concrete and general cement work silica Portland cement of a fineness such that 93 per cent. passes a 200-mesh sieve is equal to a Portland cement of which 65 per cent.

passes a 200-mesh sieve, then the following points remain to be considered: *First*, comparison between Portland cement and silica Portland cement of the same degree of fineness; *second*, economy from the consumer's standpoint, taking into consideration any results from comparison in tests.

The question at present as to preference between natural and Portland cements is in most cases not one of the unsuitability of the former, but the relative costs for the work, in view of the greater amount of the former required. This narrows the question down to the cost of each at the mill, to which is added the freight to point of consumption, and this, with cost of other materials used in concrete, forms the basis of comparison of cost per cubic yard in each case. In case of silica Portland, the following points would be involved in addition to those previously mentioned:

1. Cost of Portland at the mill.
2. Freight rate to silica Portland cement mill, which can be located near the demand for it.
3. Cost of sand.
4. Cost of unpacking and repacking, or cost of transportation of Portland-cement clinker in bulk and preliminary grinding before adding sand.
5. Cost of grinding.
6. Entire cost as compared to that of Portland cement.

MAGNESIAN CEMENT

35. Magnesian cement is a white cement that sets quickly and is by far the strongest cement known, having an estimated crushing resistance of over 1,000 tons per square foot. It is made from finely ground magnesium oxide and a solution of magnesium chloride, and owing to its cost has no ordinary commercial applications.

PORTLAND CEMENT

36. Nature and Composition.—Lime cements are essentially compounds of lime, silica, and alumina, in which magnesia and iron oxide figure to a greater or less extent. This is not strictly true, however, as in France cements have been made with alumina lacking, thus making a calcium silicate cement, but this is the exception, not the rule. Well-defined distinctions between the various classes of cements cannot be given, nor do authorities agree on an exact definition that would include all Portlands and exclude all others. An intelligent idea can only be conveyed by stating the general principles of manufacture and the chemical nature of each.

Natural cements, as the name indicates, are obtained by burning natural rock without any previous mixture or pulverizing. This, also, is only partially true, as in some cases a mixture is made by selecting broken stone from two or more veins or beds and making a rough mixture, but not the intimate or exact mixture obtained in the pulverized raw material in Portland-cement making. They are burned at a low temperature, and, while they are silicates of lime, alumina, and magnesia, the silicate ratio is never as strictly adhered to as in Portland-cement making. Other characteristics, such as chemical composition, specific gravity, tensile strength, etc., might be incorporated in a definition, but none could be framed that would be technically correct. Natural cements are low in specific gravity, tensile strength, and content of lime, and high in silica, alumina, and often magnesia.

Portland cements are made by making an accurate and uniform chemical mixturé of the raw materials, and in order to obtain this intimate mixture, it is finely pulverized before burning. With this mixture entering the kilns, a necessarily uniform chemical product is obtained if the burning is conducted properly. The percentages of the various ingredients are therefore confined to rather narrow limits and not subject to such variation as in natural cements. Portland cements are higher in lime, specific gravity, and tensile strength than natural cements. The uniformity in composition may be seen in the table following.

TABLE IX

ANALYSES OF PORTLAND CEMENTS

Mill	Silica. Per Cent.	Alumina. Per Cent.	IronOxide. Per Cent.	Lime. Per Cent.	Magnesia. Per Cent.	Sulphuric Acid. Per Cent.
Whitehall....	21.45	7.90	2.40	63.40	2.45	1.30
Alpha........	22.62	8.76	2.66	61.46	2.92	1.52
Atlas	21.96	8.29	2.67	60.52	3.43	1.49
Giant........	19.92	9.83	2.63	60.32	3.12	1.13
Vulcanite....	21.08	7.86	2.48	63.68	2.62	1.25
Dyckerhoff ..	19.35	7.00	4.50	63.75		
Germania....	21.14	6.30	2.50	66.04	1.11	

37. To obtain these results after burning, the manufacturer may use either of the following: a mixture of high-grade limestone and clay or high-grade and low-grade limestones, the latter being called *cement rock*, or, in the Lehigh district, *bluestone.* The limestones used vary from 88 to 98 per cent. calcium carbonate where two stones are used, but with clay it is customary to use only high-grade limestone. The so-called *cement rock* varies in composition, but usually runs between 60 and 75 per cent. calcium carbonate. Table X gives analyses of materials used at several places.

38. Calculation of Cement Mixtures.—This task is one of the duties of the chemist. After determining the composition of the raw materials, it is his place to calculate the weights of each to be used, in order to produce a cement having a composition similar to one of those given in Table IX. It is also his business to determine whether it should contain 61 per cent. or 63 per cent. of lime, this being based on the relative amounts of silica and alumina. Having determined the desired composition, it is customary to figure the total calcium carbonate necessary for the raw-material mixture, and this usually falls between 75 and 76 per cent., but occasionally a little higher. If 75 per cent. is to

TABLE X

ANALYSES OF MATERIALS USED FOR THE MANUFACTURE OF PORTLAND CEMENT IN THE UNITED STATES

Locality and Material	SiO_2	Al_2O_3	Fe_2O_3	$Al_2O_3 + Fe_2O_3$	$SiO_2 + Al_2O_3 + Fe_2O_3$	CaO	$CaCO_3$	MgO	$MgCO_3$	Alkalies	Loss on Ignition	Organic Matter and Loss
Penna. (Siegfrieds):												
Cement rock..........	17.50	7.70	1.50				69.80		3.97			
New Jersey (Alpha):												
Cement rock........ .	15.05	9.02	1.27				70.01		3.96			
Ohio (Wellston):												
Limestone...........	3.53			1.14		54.45		1.14				
Clay..................	69.49			16.42		2.29		.78			5.43	
Arkansas (White Cliff):				Water								
Chalk...............	6.07	.62	2.33	8.51			81.48		.96			
Clay.................	74.54	14.47	1.79			.73		.34		1.89	6.24	
Michigan (Bronson):												
Marl.................					4.00		86.00					10.00
Clay...	62.00	20.00	8.00			.50		1.00				8.00

be his standard, he checks his composition at frequent intervals, to maintain it at this point by increasing or decreasing the amount of limestone. The following example will illustrate the method of calculating a mixture of two new stones where calcium is present as carbonate or a stone and clay if lime is calculated to carbonate.

Example.—Given, a cement rock 70 per cent. calcium carbonate and a limestone 95 per cent. calcium carbonate. What proportions should be used to produce a 75-per-cent. calcium-carbonate mixture?

Solution.—Let x = weight of limestone;
y = weight of cement rock;
a = percentage of $CaCO_3$ in the stone = 95%;
b = percentage of $CaCO_3$ in the rock = 70%;
c = percentage of $CaCO_3$ in the mixture = 75%.

Then,
$$c = \frac{ax + by}{x + y};$$

or,
$$x(a - c) = y(c - b)$$
$$\frac{x}{y} = \frac{c - b}{a - c};$$

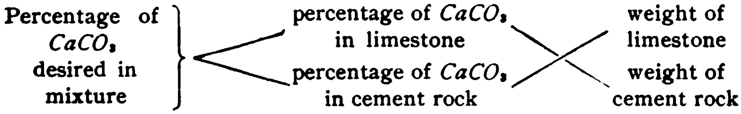
or,
$$x : y = (c - b) : (a - c);$$

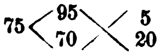
or, substituting,
$$x : y = (75 - 70) : (95 - 75);$$

or,
$$x : y = 5 : 20;$$

or, in proportions of 1 to 4. In other words, if 75 per cent. of calcium carbonate is desired, every unit of stone has 20 per cent. available or excessive carbonate and every unit of rock a deficiency of 5 per cent.

For rapid calculations on this basis, the following diagram has been devised to simplify figures:


Percentage of $CaCO_3$ desired in mixture
percentage of $CaCO_3$ in limestone
percentage of $CaCO_3$ in cement rock
weight of limestone
weight of cement rock



By use of this diagram, two simple subtractions show at once the relative proportions required. It is quite satisfactory in working with materials where all the calcium exists as carbonate, but otherwise it is not applicable unless the lime is calculated as such. The above formula, it will be observed, is only applicable to maintaining or determining a mixture that will contain a certain amount of calcium carbonate, but it does not determine whether the mixture should be 75, 76, or 77 per cent.

39. On this point, the work of Le Chatelier has been the foundation of much of our modern theory of cement. According to him, the setting of a cement when mixed with water is caused by formation of a calcium monosilicate and calcium hydrate as follows:

$$2(3CaO,SiO_2) + 9H_2O = 4Ca(OH)_2 + 2(CaO,SiO_2),5H_2O$$

On this basis, it was assumed from formulas deduced by Le Chatelier that the total basic constituents of any cement expressed in equivalents should not be greater than three times the total acid constituents also expressed in equivalents. The equivalents are found by dividing the percentage of the constituent in the cement by its molecular weight. Stated simply, the proportion of acid to basic ingredients should be such that none of the bases are left unsaturated.

40. Following in the lines of Le Chatelier, S. B. and W. B. Newberry made a number of experiments, and in their "Constitution of Hydraulic Cements" they deduce the following results and a rule, which is well adapted to cement work in many cases. Assuming that the trisilicate and the dialuminate of calcium in varying proportions are the essential constituents of cement, we have

$$x(3CaO,SiO_2) + y(2CaO,Al_2O_3)$$

in which x and y are variables whose value depends on the relative amounts of silica and alumina in the raw materials used. The formula $3CaO,SiO_2$ corresponds to 2.8 parts, by weight, of lime to 1 part of silica and is shown as follows:[*]

$$CaO = 56,\ 3CaO = 168,\ SiO_2 = 60,\ \tfrac{168}{60} = 2.8$$

---

[*] For convenience in all calculations, atomic weights are taken as follows: $Ca = 40$, $O = 16$, $Al = 27$, and $C = 12$.

In the same way, $2CaO,Al_2O_3$ corresponds to 1.1 parts, by weight, of lime to 1 part of alumina:

$$2CaO = 112,\ Al_2O_3 = 102,\ \tfrac{112}{102} = 1.1.$$

They substitute weight for equivalents and obtain the following rule for showing the maximum lime for a good cement:

To find the number of parts of lime for 100 parts of clay of known composition, "multiply the percentage of silica by 2.8 and the percentage of alumina by 1.1 and add the products; the sum will be the number of parts of lime required per 100 parts of clay."

Or, let x = parts of lime required for 100 parts of clay;
a = percentage of silica in the clay;
b = percentage of alumina in the clay.

Then, $$x = 2.8\,a + 1.1\,b. \qquad \textbf{(1.)}$$

Figuring lime to calcium carbonate, 2.8 becomes 5, ($CaCO_3 = 100$, $CaO = 56$, then $\frac{100}{56} \times 2.8 = 5$), and 1.1 becomes 2 ($\frac{100}{56} \times 1.1 = 2$).

Let x' = parts of calcium carbonate required for 100 parts of clay;
a = percentage of silica in the clay;
b = percentage of alumina in the clay.

Then, $$x' = 5\,a + 2\,b. \qquad \textbf{(2.)}$$

EXAMPLE (*Newberry*).—Composition of clay is as follows:

Silica SiO_2	65.4%
Alumina Al_2O_3	16.5%
Ferric oxide Fe_2O_3	6.1%
Lime CaO	2.2%
Magnesia MgO	1.9%
Moisture, etc.	7.9%
Total	100.0%

Applying formula 1,

2.8 × 65.4 per cent. SiO_2	= 183.12
1.1 × 16.5 per cent. Al_2O_3	= 18.15
Sum	= 201.27
CaO in the clay	= 2.20
Parts of CaO required for 100 parts of clay	= 199.07

As 56 parts of CaO correspond to 100 parts of $CaCO_3$, the amount of $CaCO_3$ required for 100 parts of this clay are:

$$199.07 \times \tfrac{100}{56} = 355.5 \text{ parts of } CaCO_3.$$

This gives a mixture containing 78 per cent. of $CaCO_3$ and 22 per cent. of clay.

41. The diagram in Art. **38** is a convenient method of getting a mixture when the percentage of calcium carbonate has been determined. In other words, if by experience or otherwise it has been shown that 75 per cent. of calcium carbonate makes a good cement of materials of a given locality, the diagram is a short cut; but if it is a new locality or a new chemist takes hold of an old locality, Newberry's formula determines the proportions, and from this the percentage of calcium carbonate can be calculated. The advantage of the diagram is that it gives rapidity in calculation, but it is a simple formula, taking into account nothing but calcium carbonate, and in localities where silica, alumina, and iron are subject to slight variations only it is quite satisfactory after a standard has been determined, whereas the Newberry formula is adapted to all kinds of materials adapted to use in cement, and to all variations in lime, silica, and alumina within the limits in those materials. There are exceptions to this, however, as while the formula may call for 77 or 78 per cent. of calcium carbonate in a mixture, experience may show that it gives a cement with an excess of lime and liable to check or warp on boiling test, as described later. In such case the percentage of lime must be reduced.

PROCESSES OF MANUFACTURE OF PORTLAND CEMENT

42. Having seen in the preceding pages the principles on which the manufacture of Portland cement depends, the methods of obtaining these results fall under two heads: (1) Rotary-kiln process (dry-mix, slurry); (2) set-kiln process.

43. Dry Rotary-Kiln Process.—The **rotary dry process** is by far the more important and will be discussed at some length. The *raw materials*, assuming that limestone and cement rock are used, are brought to the crushers, which may be gyratory, jaw, or roll, the former being the most common, and crushed separately so as to pass a 1-inch or a 1½-inch ring. It is then conducted to separate bins until ready for mixing; or, in some cases, the mixture is made before crushing and the raw material passed through various stages of grinding to kilns without further handling. The mixture is then calculated by means of the foregoing formulas, and the proper amounts of each material weighed upon a car or barrow and conveyed or elevated to the drier.

44. Drier.—This consists, as shown in Fig. 6, of a cylinder *A* of boiler iron, usually about 40 feet long and 4 feet in diameter, and is placed in a nearly horizontal position, the feed end being elevated slightly. In the figure the elevator buckets *b* will be seen at the rear end, just behind the stack *c*. This elevator discharges into a chute that leads material under the stack and into the higher end of the drier. From the motor, gearing, and tires riding upon idlers, it will be observed that the drier is constructed to revolve on its axis and is geared to rotate about one or two revolutions per minute. On the inner surface of the cylinder, a number of pieces of 4-inch angle iron are fastened parallel to one another and to the axis of the drier. The front end of the cylinder extends into a masonwork chamber, as at the rear; this chamber consists of a fireplace, the flue being the drier itself, the gases of combustion passing the entire length and up the stack at the rear.

The crushed stone fed in from the elevator gradually works its way forwards, owing to the rotation of the cylinder, the angle irons carrying it up the side and dropping it off as it turns over, thus mixing it thoroughly, and as it works forwards it is dried by hot gases passing in the opposite direction and is discharged at the lower end dried and mixed, ready for finer pulverizing. To reduce the raw material still further, various mills may be used.

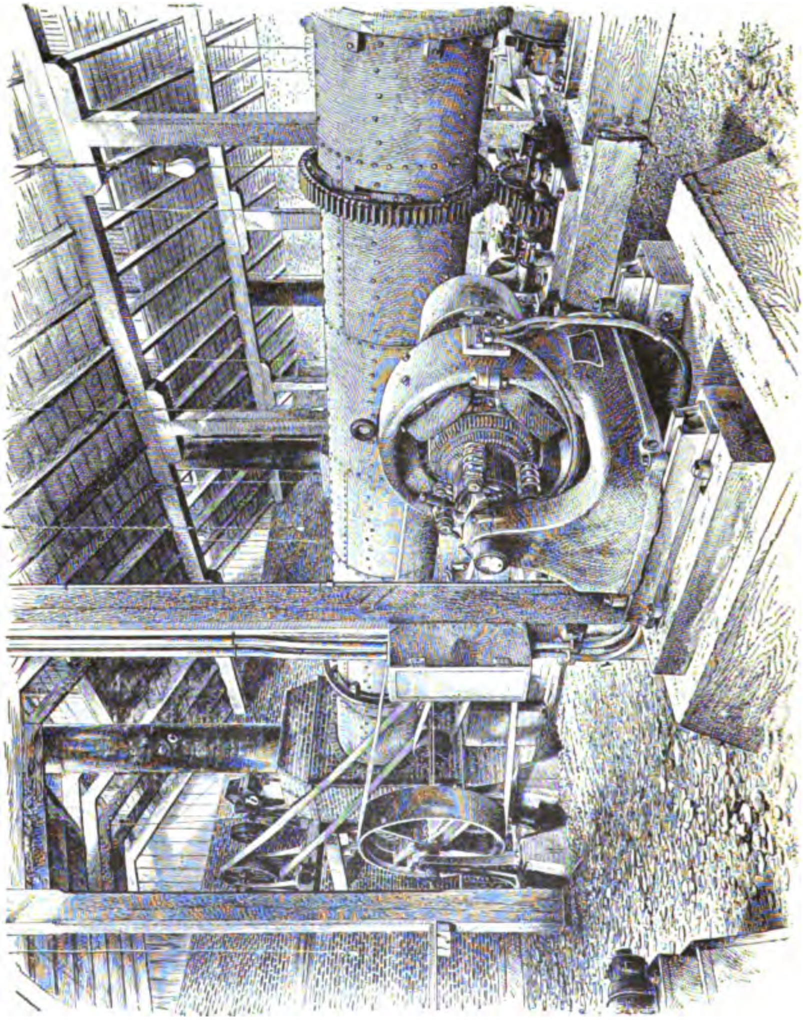


FIG. 6

45. Pulverizing.—Ball mills and tube mills are coming into quite extensive use. These are worked in batteries or pairs, the ball mill doing the coarse grinding and the tube mill the fine pulverizing.

46. Ball Mill.—Figs. 7 and 8 show a **Krupp ball mill,** which consists of a steel drum *a* about 8 feet in diameter

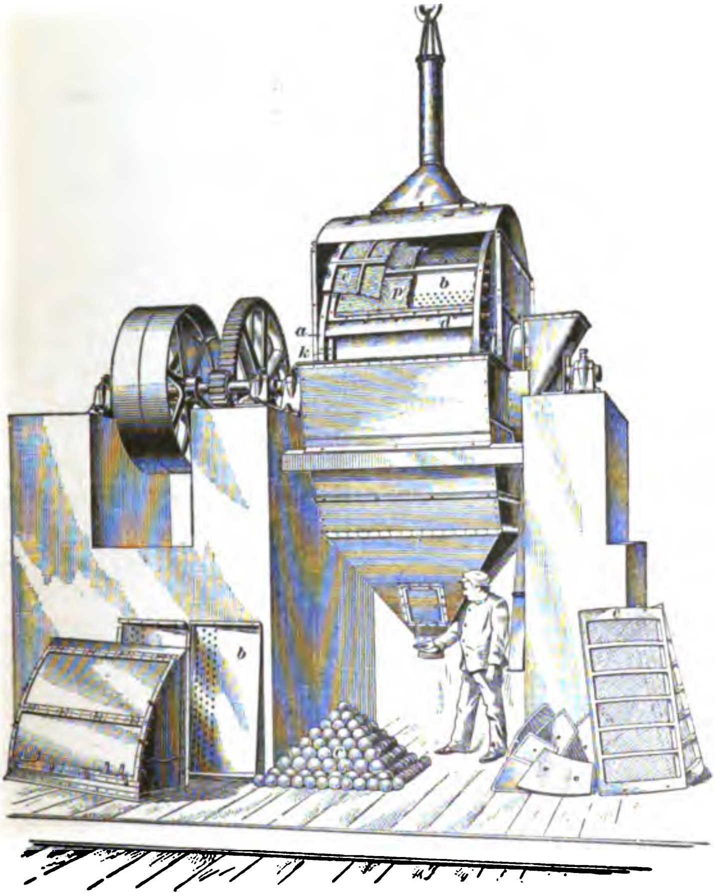


FIG. 7

and 4 feet wide, the inner surfaces of which are lined with heavy plates of tough and hard steel. The drum revolves upon a central axis and carries with it part way round a number of steel balls *c*, which are made to roll or fall upon the material to be crushed by a series of offsets or steps *h* in the grinding plates. At each of these offsets is a slot, or opening, for the return of material to the mill. It should first be observed that the grinding plates *b* are perforated, and the balls grind, or force material through to the outside, where it works its way to a series of fine screens *e* and *p* that separate the fine particles by allowing them to pass to the outer casing and drop to the hopper below, while the coarser particles are returned by scoop plates *d* through slots, or return channels, to the mill for further reduction. The fineness is regulated by the number of meshes in the outer screen *e*, which is usually from 12 to 20 per linear inch. This screen is bolted by setscrews to the rim *a*, the coarser and inner screen *p* being bolted to the rim *k*. Such screens do not furnish material in a sufficiently fine state, but with this for preliminary grinding it is then put through a "grit," "pebble," or "tube" mill, as it is variously called.

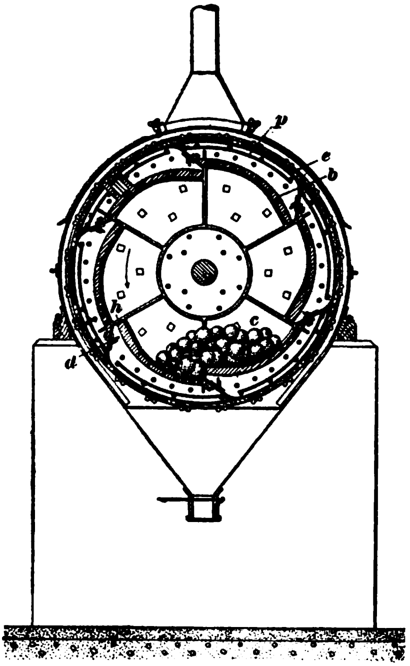


FIG. 8

47. Grit, or Tube, Mill.—This consists of a cylinder of boiler plate usually 5 feet in diameter and 22 feet long, suspended upon trunnions, or bearings, at either end, and rotating about a horizontal axis. The cylinder is lined with flint blocks cemented in, or tough cast steel, or other metal plates, and filled to about half its capacity with hard Greenland or Danish flint pebbles varying from 1 inch to 3 inches in diameter. By an arrangement of a screw conveyer at

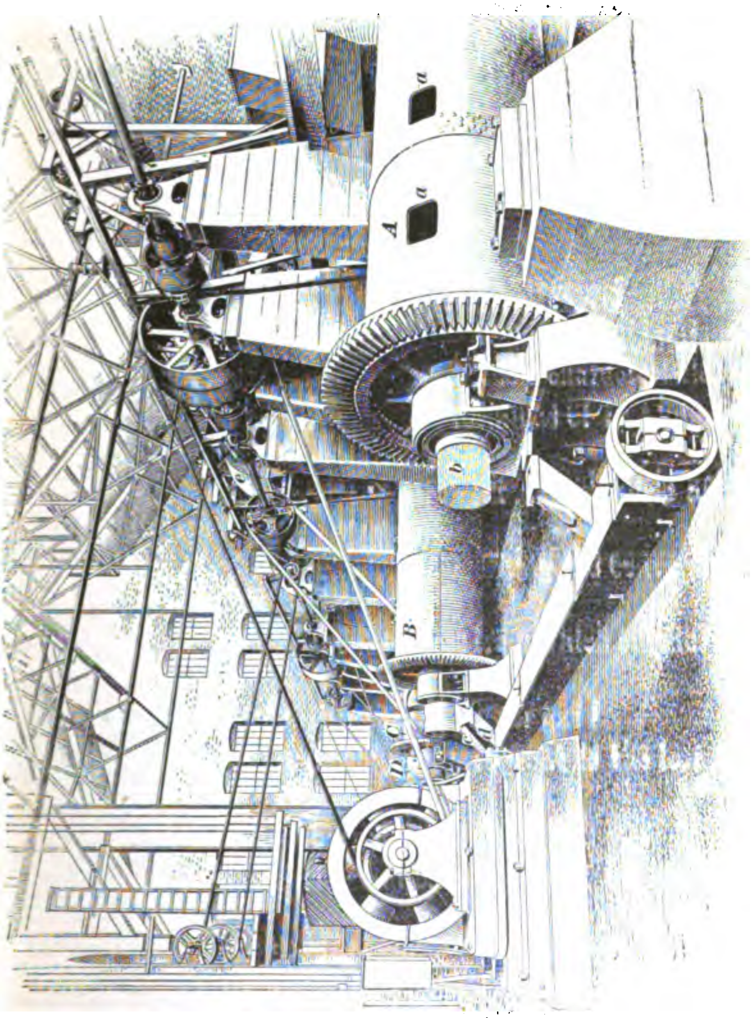


FIG. 9

one end and sometimes both ends, material is fed in and discharged at points lying in the axis of the cylinder. The material to be pulverized is fed in at one end and works its way towards the other, while the mill is kept constantly rotating at a rate of 25 to 28 revolutions per minute. The tumbling of the pebbles upon one another, with the material to be ground forming the bed in which they roll, causes the fine pulverizing action of the mill. The fineness is regulated by a device at the feed end of the mill, which can be set to control the amount of material entering, and this in turn regulates the speed of material passing through and, consequently, the fineness to which it is reduced. By this mill, a material 95 per cent. of which will pass a 100-mesh screen can easily be obtained.

48. Whitehall Raw-Stone Mill.—Fig. 9 shows four tube mills *A*, *B*, *C*, and *D* in parallel arrangement, but the ball mills completing the batteries can scarcely be seen on account of piers and shafting. In line of the axis of the tube mill *C*, third from the front and back of the shaft, the casing *e* of one ball mill may be seen. Material is discharged from the ball mills into hoppers over the tube mills. The tube mill *A* in the foreground is partially dismantled, showing manholes *a* for charging pebbles and ingress and egress for repairing. The casing is also removed from the discharge screen *b*, showing method of separating fine material from bits of broken pebbles occasionally discharged. The second mill *B* shows two spouts *c* and *d*, the lower one *d* for finely pulverized material and the upper one *c* for coarse particles.

49. Griffin Mill.—This mill, shown in Fig. 10, though more complicated and consisting of a much greater number of parts than either ball or tube mills, is admirably adapted to grinding or pulverizing both raw stone and clinker. It consists of a shaft *a*, vertically suspended upon a universal joint, which is composed of a ball *f*, with trunnions attached, which latter work in half boxes that slide up and down in recesses in the pulley-head casting. The shaft is driven by the pulley *j*. At the bottom of the shaft will be seen a fan *g*, which draws air in from the top of the cone *h* and throws finely pulverized material against the vertical screen *b* on its own level. Below this fan is a roll consisting of two parts, a body *k* and a tire *c*. The body of the mill consists of a base, or pan *d*, against which the roll revolves, the tire coming in contact with the steel ring, or die *e*, and it is

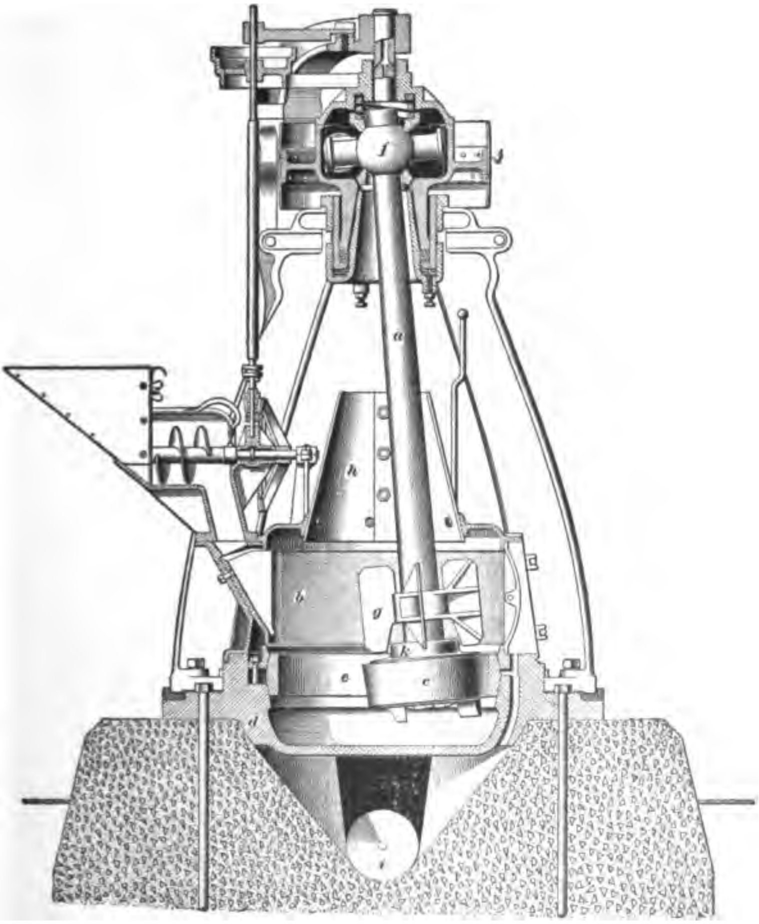


FIG. 10

between these that the material is pulverized, the finer particles being driven through the screen and the coarser ones falling to the bottom of the pan, only to be stirred up again by the plows, or scoops, on the bottom of the roll and brought between the die and tire. In mills for dry pulverizing, the base has a number of openings downwards, outside of the die, through which the pulverized material is led to the screw conveyer *i* underneath.

50. Burning.—The burning of Portland cement may be accomplished either by an intermittent or a continuous process. The former method was the earliest in use, but is now practically abandoned. Intermittent kilns may be divided into two classes, *shaft kilns* and *dome kilns*, either of which may be used as continuous.

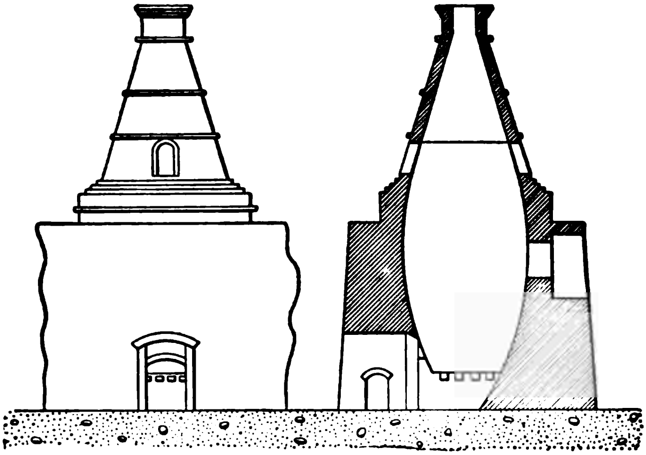


FIG. 11

51. Shaft Kilns.—Kilns of this class are similar to those commonly used in burning lime. They vary considerably in minor details of construction, but are all alike in principle. Fig. 1 illustrates a common type, the height of which may vary from 20 to 40 feet and the diameter from 10 to 12 feet. The sides may be straight, or curved as shown. In all, the method of charging is the same. For making lime, hydraulic lime, or natural cement, the raw material undergoes no preliminary preparation other than to be broken into pieces of convenient size. For Portland cement, the raw materials are finely ground together in proper proportions, mixed with water, molded into bricks, and dried. Layers of fuel and raw material are arranged alternately from bottom to top and the furnace fired.

In intermittent working, the fuel is allowed to spend itself and the entire product is drawn from the bottom when cool. This primitive process, rarely used now, even for lime, has long been abandoned, as the product was not uniformly burned.

In continuous working with the same kilns, the material is drawn from the bottom as fast as it is burned, and as the mass descends, successive layers of stone and fuel are added above. The simple shaft kiln is not well adapted to making Portland cement and was superseded by dome kilns.

52. Dome Kilns.—These are also of many types, of which the low forms may be used either intermittently or continuously. Fig. 11 shows an early English kiln still in use. It consists of a shaft from 9 to 12 feet in diameter at the widest part, and 15 to 20 feet high, which forms the body of the kiln, and which is surmounted by a chimney, or stack, from 12 to 18 feet in height. It is charged through doors shown at the side.

The German form shown in Fig. 12 resembles a blast furnace somewhat. The sides are straight, making the body cylindrical. It has a diameter of from 9 to 12 feet and a

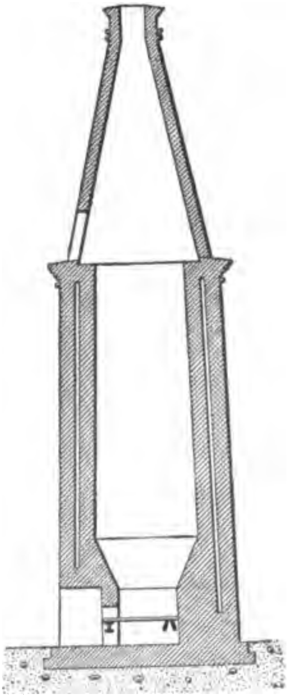


FIG. 12

height of from 25 to 30 feet. The height of the chimney is much greater than in the English kiln, and ranges from 35 to 60 feet. Fig. 13 shows a French form of dome kiln.

The form shown in Fig. 14 is known as the **Shoefer kiln** and is designed to preheat the bricks by the waste gases before they reach the combustion chamber. It consists of a tall chimney *A* and a vertical shaft *B*, which is divided into three sections. The bricks are charged through doors *e* into preheating chamber *a*, from which they descend into the narrow combustion chamber, or crucible *b*, where burning takes place, and thence to the wider section *c*, which serves to cool the clinker and heat the draft. The fuel is charged from the floor *k* below the brick-charging floor *l* through stoke holes *f*. The kiln is economical in fuel, as the waste gases passing the charging doors *e* are at a low temperature, showing that a loss of heat through the chimney

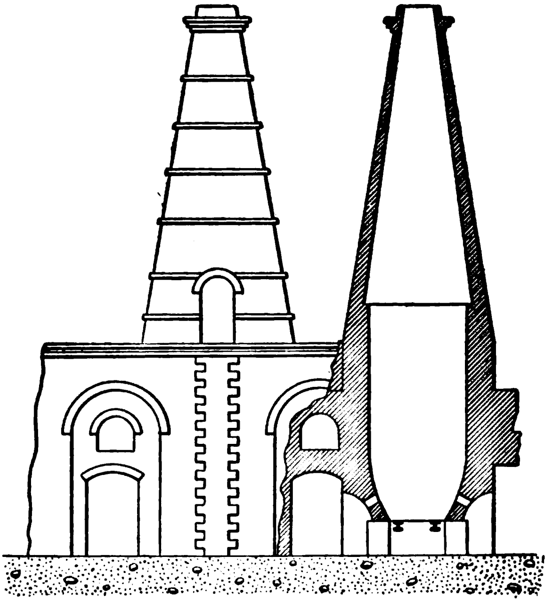


FIG. 13

is avoided, and the clinker drawn from the bottom *c* is cool, showing that the heat of the clinker has been utilized in preheating the draft.

In the use of vertical kilns, the raw material is prepared in much the same manner as for rotary kilns, but usually by a different style of machinery. The finely ground raw material is mixed with sufficient water to permit it to be molded into bricks. These are then placed upon a drying floor or in a drier and the moisture driven off until the brick is sufficiently hard and dry to permit handling. They are then taken to the kiln and charged. This is done by placing a layer of fuel, coal or coke, and upon this a layer of dried bricks, either piled carefully or simply spread over to a uniform thickness. Upon this another layer of fuel is placed, and then another of the bricks until the kiln is full. In the continuous kilns of the shaft type, the fire is kept going constantly, new layers being added to the top as the burned product is drawn off below, calcination and clinkering thus taking place as the charge descends. In the dome kiln, it is customary to make the entire charge at one time and then, closing the charging door, to calcine and clinker the entire contents at one operation. Various modifications may be used, but in any case of vertical kilns the same objections are to be met—namely, cost of labor, as most of the work must be done by hand, and cost of drying.

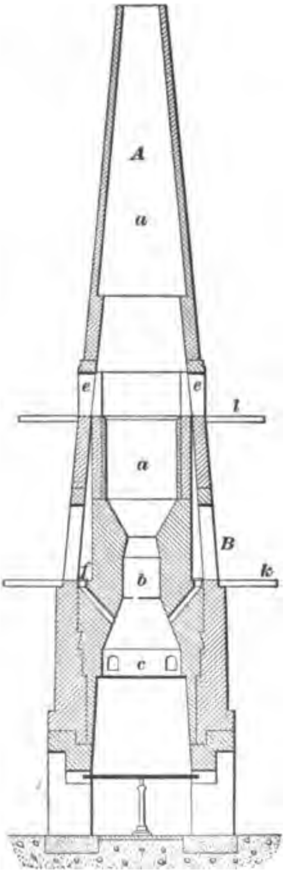


FIG. 14

The objections mentioned apply to continuous kilns, which, in addition, are expensive in construction and require skilled labor to operate. They are, however, economical in fuel consumption, which is usually about 10 or 12 per cent. of the weight of the clinker burned. They are in common use in Europe, but have not found much favor in this country.

The Hoffman kiln, another form of continuous kiln, is also economical in fuel, but open to the same objections as the others, and while used quite extensively in Europe, has not been adopted in the United States.

53. Rotary Kiln.—About 90 per cent. of the Portland-cement mills in this country use **rotary kilns**, and in the past few years no other kind has been constructed. The kiln was invented by Siemens in 1865 and improved by Ransome in 1885, since which time various modifications in dimensions and method of driving have been made.

Fig. 15 shows a modern kiln room equipped with ten rotary kilns *A*. As now used, the kiln consists of a slightly inclined cylinder, varying from 45 to 70 feet in length and 5 to 7 feet in diameter, the most common size being 60 feet long and 6 feet in diameter. The outer casing or shell is made of boiler steel, and this is lined with refractory firebrick ranging in thickness from 9 inches at one end to 3 or 4 inches at the other, or it may be of uniform thickness. The upper end projects into a dust chamber *b* connected with stack *c*, but, in order to permit rotary motion, is not in contact with the masonry. Above the dust chamber is a convenient size storage bin *D*, from which a screw conveyer leads the finely pulverized dry-cement mixture to an inclined feedpipe leading to the kiln. This feedpipe is protected from the heat of the issuing gases by a water-jacket. The kiln is so geared that its speed can be regulated between one revolution per minute and one-third of a revolution per minute by means of a variable speed controller. As the kiln revolves, the material works its way slowly down the incline towards the front end, which, while open, is protected by a hood or shield *e* of firebrick placed very close to the end. Through this hood the fuel is introduced by means of the pipe *f*.

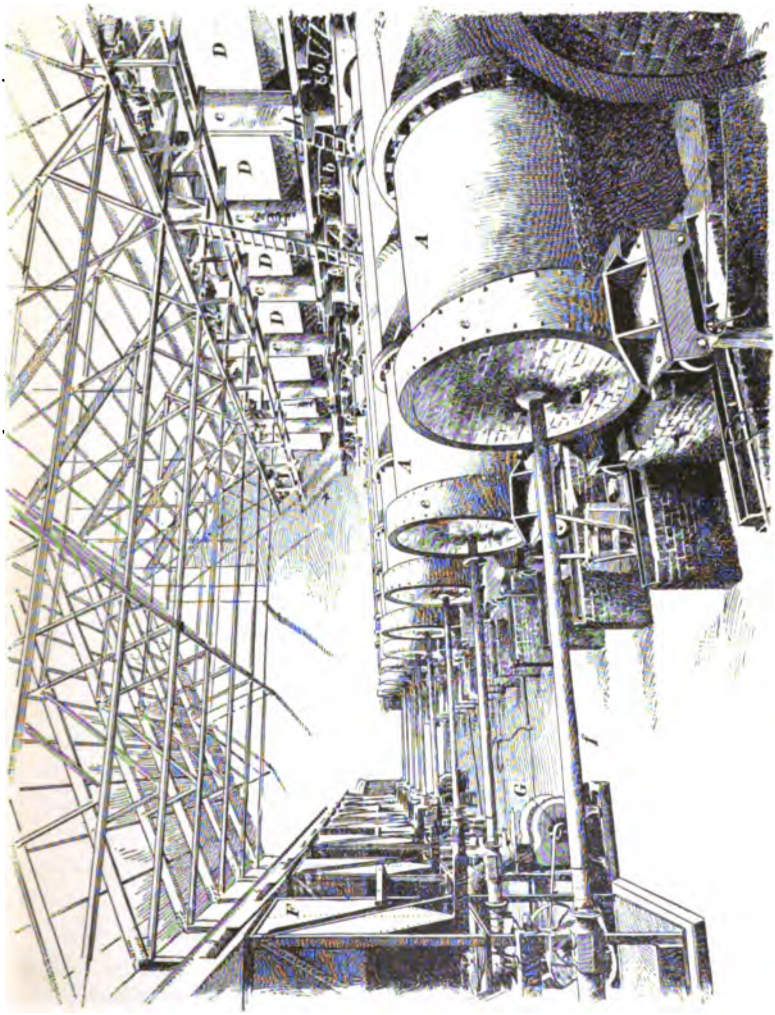


FIG. 15

54. Fuel.—Formerly the fuel used was oil blown in by a jet of steam or compressed air, but this, becoming too expensive, was abandoned and powdered bituminous coal, preferably gas slack high in volatile matter, was used instead; experiments have been made using producer gas. Coal is the fuel most commonly used, and this is pulverized in much the same way as raw material, using either the Griffin or tube mill to reduce it to a state that 92 or 94 per cent. will pass a 100-mesh screen. The pulverized coal is stored in convenient bins *F* near the kilns and is blown through the pipe *f*, through the hood, by jets of steam and compressed air, or more commonly by air from a fan *G* producing a pressure of 1½ to 3 ounces per square inch. Very active combustion takes place on entering the kiln and produces almost a white heat or a temperature of about 2,800° F. The gases of combustion pass the entire length of the kiln into the dust chamber *b* at the rear and from thence to the stack. The temperature of gases leaving the kiln at the point where the finely pulverized cement material enters ranges from 900° to 1,200° F.

55. Changes Produced by Burning.—The first stage in burning the cement consists of calcination, by which is meant driving off the carbon dioxide from the carbonates present. The material works its way forwards into the region of more active combustion, and here reactions between silica, lime, and alumina take place. Exactly what these reactions are is difficult to say, as there is a wide difference of opinion. All agree, however, that the results of these reactions are silicate and aluminate of calcium, but in what proportions or what formulas should be ascribed to them is still open to discussion. The effect of heat on the chemical composition and the solubility of the mixture is easily determined.

If a sample of raw material be taken as it enters the kiln, it will be found that all of the lime and magnesia and part of the alumina and oxide of iron are soluble in strong hydrochloric acid. All of the silica and part of the alumina and iron are insoluble in the acid. These proportions vary according to whether the raw mixture is a natural Portland-cement rock—that is, a marl or argillaceous limestone of about 75 per cent. calcium carbonate or only slightly corrected by limestone or clay—or whether the raw material is a mixture of two dissimilar materials, as limestone and clay or chalk and clay. In the former case, the soluble portion may be high, while in the latter nearly all the clay (which here would be 25 per cent. of the whole raw material) may be insoluble. In one particular case, 21 per cent. of the whole raw mixture was found to be insoluble. A chemical analysis of either would require a fusion to separate all the silica and alumina.

If a sample of the burned cement be analyzed, it will be found that it is practically all soluble in hydrochloric acid. In fact, solution is so complete that the majority of cement chemists do not regard a fusion necessary in analysis of a cement. In the kiln, therefore, reactions must take place that rearrange the combinations in which silica and alumina, the insoluble ingredients, are held. In doing this, it also makes new combinations with the lime and, in fact, readjusts the chemical bonds all around. In the raw material, silica exists as free silica (sand) or as silicate of alumina, both of which are insoluble, while alumina exists chiefly as silicate of alumina and, as just stated, is insoluble. In this case, alumina acts as a base and is combined with silicic acid, forming a silicate. In the presence of a large amount of a base, alumina acts as an acid instead of a base and forms an aluminate. In this case, the base is lime, and silica and alumina act similarly (as acids) and form silicate and aluminate of calcium. These are both soluble in acids. The change of alumina as a base in the raw material to an acid in the cement and the consequent change in solubility is brought about by the action of heat.

That this action does not take place by mere calcining without clinkering is shown by the following example. Samples were taken, after it had cooled, from a 60-foot kiln suddenly stopped by accident. At the flue end the composition of the material showed the normal composition of raw mixture and, consequently, about 33 per cent. of carbon dioxide. In the middle, the material showed a very small amount and 40 feet from the flue end practically none. The material, however, was still in the form of powder of a yellowish-brown color and showed relatively about the same amount of insoluble matter as in the uncalcined mixture. Five feet distant (at 45 feet from the flue end) fusion had begun and the amount of insoluble matter fell off 50 per cent., at 50 feet it had fallen off 90 per cent., and at the 55-foot mark the material had all fused to a clinker containing no insoluble matter, which showed conclusively that alumina no longer existed as simple silicate of aluminum. It might be considered as a double silicate of calcium and aluminum, but authorities generally agree in calling it aluminate calcium. As to the quantivalent ratio of the silicate of calcium, there is no consensus of opinion. Some claim it to be a straight trisilicate, others a mixed bisilicate and trisilicate and others a double silicate. Whatever the reaction may be, it is certain that it changes the silica and alumina into a form in which all is soluble, and, moreover, this does not take place at a low heat, but only when the temperature is sufficiently high to cause fusion, thereby bringing the lime into close enough contact with silica and alumina and under conditions favoring the assertion of the chemical affinity between them. Analysis of hydraulic limes burned at a lower temperature shows a very large proportion of the silica and alumina insoluble. The one very great distinguishing feature, then, between hydraulic limes and hydraulic cements on one hand and Portland cement on the other is the entire reconstruction of the chemical bonds in the latter as against the partial reconstruction in the former. The beneficial effects of magnesia in natural and the deleterious effects in Portland cement are demonstrated to be due to the different temperatures at which the cement is burned.

56. Returning, now, to the process of burning, the mass is first calcined and then brought to incipient fusion, the pulverized material forming small kernels from the size of a pea to that of a walnut or even larger, and when properly burned they are of a greenish-black color, frequently carrying small yellow spots in their interior. Great care must be exercised in burning, and it is done by men specially trained for this work. If the cement is not sufficiently burned, several per cent. of carbon dioxide may remain and the resulting kernels, or clinkers, as they are called, may be light and porous and produce a poor cement when ground. If cement is overburned, the kernels usually have a decidedly yellow color in their interior, and while it is a disputed question whether this affects the cement or not, it is, nevertheless, to be discouraged, as it carries with it a waste of fuel and producing, as it does, a harder clinker than necessary, adds to the cost of pulverizing. Excessive overburning seems to produce a cement low in tensile strength and with impaired setting properties, but moderate overburning is a lesser evil than underburning.

57. The disposition of hot clinker dropping from the end of the kiln varies in different mills. In some, the kiln room is on a higher floor than the rest of the mill and the clinker is dropped into pits and allowed to cool. In others, it is spread on the floor or conveyed to a heap; while in the most modern mills it is carried by a bucket elevator to the top of a cylindrical sheet-iron tower 30 to 40 feet high and about 8 feet in diameter, the tower being kept full at all times and a blast of air from a fan kept passing through it from the bottom upwards. As the clinker cools, it is drawn off at the bottom and carried to the mills, but in any case, whether towers are used or not, it is never ground while hot, as it clogs the screens on the mills.

58. Slurry Process.—The slurry process is a combination of the old set-kiln method of preparing raw mixture with the rotary system of burning. It is in use in a number of plants in the Middle West, where soft, wet marl and clay are the raw materials used. The marl is found in beds from 20 to 30 feet thick in the lowlands, where it was deposited under water, and instead of a crystalline texture, it is amorphous and so fine and uniform that, without any preliminary crushing, 90 per cent. of it will pass a 20-mesh screen. As taken from the bed it contains about 40 per cent. of solid matter and 60 per cent. of water. At Bronson, Michigan, the analysis of solid matter is as follows: Calcium carbonate, 86 per cent.; silica, alumina, and oxide of iron, 4 per cent.; organic matter, 10 per cent. This mixture of marl and water is taken up by a dredge and placed in a pug mill or clay mixer and mixed with water from a small pump on the dredge. After being thoroughly mixed, it is allowed to run to the sump of the pump, which delivers it to a pipe line and thence to the mill, where it is stored in large basins. Clay is brought to the mill and stored on a floor above the grinding mills. When the mixture is made, it passes to the edge-runner mills, which are similar in principle to the dry pan. They consist of vertical wheels with broad faces upon a horizontal axis, and grind by means of these faces crushing the material upon a horizontal pan or bedplate, as they roll. It is further pulverized by passing through wet grinding mills, which may be of several types. In this case they consist of flat, iron disks revolving against one another, the wet material being fed through a hollow shaft at the center and discharging at the periphery into another basin. From here it passes to steel dosage tanks, where it remains until its composition is determined in the laboratory. If this is not normal, it is corrected by the addition of marl or clay, after which it is ready for burning. Up to this point the process is the same as for the set-kiln slurry process, where the mix is cut into bricks, dried, and burned in a shaft kiln. In the rotary process, this wet mix is pumped into the rear end of the kilns, where, after the 50 per cent. of the water it contains is driven off, the process is identical with the dry rotary process.

59. Final Pulverizing.—The process of finishing a cement is similar to that of grinding raw materials, and may be done by a Griffin or other gyratory mill or by a battery of a ball and a tube mill. In some of the older plants, burrstones or millstones are used, but in modern mills they have been abandoned.

60. Elevators and Conveying Machinery.—Machinery for handling raw materials, partially finished and finished product in cement mills, while similar to that for handling stone, ore, grain, etc., has necessarily some special modifications. Crushed stone and clay from quarries may be elevated to crushers in cars or buckets and, in the latter case, either in inclined or vertical elevators, the buckets being attached to iron or steel link belts or to leather or rubber belts. The same style of elevators are used at all other points except where hot clinker is carried, and here it is necessary to use link-belt elevators. For carrying coarse material, belt conveyers are admirably adapted in some cases, while for carrying fine material, either screw, belt, or scraper conveyers may be used. Screw conveyers range in size from 9 to 16 inches in diameter, upon a shaft of about $2\frac{7}{16}$ to $2\frac{15}{16}$ inches in diameter, the entire conveyer being enclosed in a box or casing. Belt conveyers carry their load on the surface and are rarely enclosed, while scraper conveyers (belts with paddles or angles attached) drag the material in an enclosed box or casing. Each has its advantages and disadvantages, which are only to be learned by actual observations in their applications in mill practice. This subject is of greatest importance in economy in production. In modern mills, it is the aim to avoid manual labor in handling material from the time it leaves the quarry to the time it leaves the stock house for shipment.

MANUFACTURE OF CEMENT

(PART 2)

TESTING OF PORTLAND CEMENT

1. Introductory.—**Tests of cement** may be classified as **physical** and **chemical.** It is largely upon the results of the former that the value of a cement is determined; not only because in actual practice it is subjected to a physical strain, but also from the fact that no special laboratory or large amount of apparatus is necessary. Moreover, it does not require as much previous training as is necessary to fit one for making chemical analyses. It must not be inferred, however, that simple as the directions may seem, any one can become an expert tester in a few days. Time and practice only can make a skilful physical tester, as it has been repeatedly shown that the same cement in the hands of two different testers may give very widely differing results. In fact, some very high-grade cements have been condemned on many occasions, owing to a lack of experience on the part of the tester; or, owing to the neglect of a skilful operator to observe some of the numerous precautions necessary to assure uniform results.

PHYSICAL TESTS

2. Fineness.—Fineness is generally determined by sifting upon 50-, 100-, and 200-mesh sieves. By this is meant sieves having that number of meshes per linear inch. The size of each of the meshes depends on the gauge of the

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wire, which, for the 50-mesh, should be about .21 millimeter in diameter; for the 100-mesh, about .12 millimeter; and for the 200-mesh, about .06 millimeter. On the finer sieves there is great difficulty in getting uniformity of results, owing to the stretching of the gauze as it is put on the frame and the consequent variations in the size of the mesh. In coarser screens, shaking to and fro is all that is required, but on the finer ones it is necessary to jar the sieve frequently against something.

It is generally conceded that the finer the grinding, the more active is the reaction in setting, though a fine cement is not necessarily quick setting. While a very finely ground cement, neat, may not develop as high a tensile strength as a coarser one, it usually exceeds the latter in sand tests, and as this is more nearly the test in actual practice, it serves as a better indication. The cementitious value, or adhesive power, of a cement depends on its fineness, and it is claimed that the higher results on neat tests with coarse cement is due to the fact that the coarser particles act as sand, thereby combining both cohesive and adhesive effects. However this may be, it has been conclusively shown by a series of tests on the same cement, some samples of which were coarsely ground and others finely ground, that the latter showed greater adhesive power—that is, gave higher sand tests—while the former showed greater cohesive power, as shown in neat tests.

Specifications as to fineness vary, depending on the judgment of the engineer in charge of the work, but of late years the tendency has been to demand finely ground cements. At present, a cement of which 94 per cent. will pass a 100-mesh sieve and 78 to 80 per cent. a 200-mesh sieve is considered satisfactory upon this point. For determination of fineness, the sample may vary from an ounce to a pound, depending on the means of shaking, whether by hand or machinery, and the accuracy of the balance used. For laboratory work, a sample of from 10 to 50 grams is sufficient.

3. Setting Time.—By this is meant the time of hardening when mixed with water. The amount of water used is generally about 20 per cent. of the weight of cement, but it may be several per cent. more or less, depending on the cement in question. Several ounces of the cement are weighed out and placed upon a glass or other non-absorbent plate and the desired amount of water added. It should be noted that both cement and water should be about the same temperature—65° to 75° F. After water is added the mass is thoroughly mixed by working with spatula, trowel, or hands in rubber gloves until water begins to come to the surface. It is then packed tightly in a mold and struck off level with a trowel or spread in a thin cake upon a glass plate and the time of hardening, or "setting," noticed. For this purpose it is customary to use Gilmore's needles. One of these is a needle about 6 inches long and having a section of $\frac{1}{12}$ inch and a weight upon it of $\frac{1}{4}$ pound, while the other has a diameter of $\frac{1}{24}$ inch and a weight of 1 pound. When the cement is sufficiently hardened so that the former weight gently applied to the surface makes no impression, it is said to have its *initial set;* and when the heavier weight makes no impression, it is said to have its *ultimate*, *hard*, or *final* set.

4. Quick-Setting and Slow-Setting Cements.—A cement that takes its initial set in less than 45 minutes and its final set in from 1 to 2 hours is called a **quick-setting**, and one that requires from 1 to 3 hours for the initial and from 3 to 8 hours for the final set is called a **slow-setting cement.** As a rule, only slow-setting cements are desired, as they generally develop higher tensile strength than quick-setting cements; but for certain kinds of work a quick-setting cement is desired and specified. In such a case, however, a minimum setting time is usually named at 15 minutes, as a cement may be so quick setting that it is impossible to get it in the mold, and such should be condemned, since it indicates an inferior article.

Quick setting may be ascribed to three causes: (1) To low lime working up soft and very smoothly and setting with a "flash"; (2) to improper mixture of the retarder (this will be discussed later, under the heading "Gypsum");

(3) to underburning, as the hydration of the loosely combined lime evolves much heat and hastens reaction.

5. Indications of Setting.—If a cement be mixed and worked properly, as directed under the heading "Tensile Strength" in the following paragraphs, it will be noticed that after mixing the surface is smooth, shiny, wet, and plastic. After a period of time, the length of which depends on the nature of the cement, it will be observed that the water disappears, it loses its luster, and the mass becomes less plastic.

According to Le Chatelier, the setting is due to a deposit of interlocking crystals from a supersaturated solution, part of the water entering into chemical combination. The length of time elapsing between mixing and the point when crystallization has advanced to such a point that it loses its plasticity and the lighter of the Gilmore needles makes no impression, is called its initial set. In quick-setting cements, this is distinctly marked, but in slow-setting cements the transition from the plastic to the initial stage is frequently so gradual that no definable initial set can be given.

6. While the importance of judging a cement by its initial set must not be underestimated, it is sufficient for all practical purposes in the case of slow-setting cements to come within ½ hour, or even 1 hour, of the actual time. For instance, if specifications call for a slow-setting cement, it is usually immaterial whether the initial set is in 1 hour, 2 hours, or even 3 hours. It is customary to specify only a minimum length of time for the initial set. If quick setting is desired, both maximum and minimum times are fixed. The former is usually about 45, and the latter 15 minutes. The minimum time is fixed; because a cement that sets in less time (some even set before they can be put into the mold) hardens before the reactions are complete, and poor tests follow. A more important consideration than the failure of the tests is the fact that unless worked up in small quantities it is impossible to use it for the work intended, and if put in, the work is very likely to prove defective in time.

7. Primary and Secondary Sets.—These may be either **initial** or **final.** A cement may take its hard set in about 8 hours and the hardening continue for several months or even an indefinite length of time, and if it then be reground and gauged again, it will be found to have setting properties; but the reactions will be less energetic than in the first case, as will be observed by a comparison of tensile-strength tests on the original and the reground cements. This is merely a point of interest; it is of no value either to manufacturer or consumer, as cement is never reground and used again.

The primary and secondary initial sets, however, are frequently a source of much annoyance both to the manufacturer and the tester, who subsequently passes judgment on it. When a cement is mixed with water, it is certain that, whatever they may be, chemical reactions at once take place, and when these have progressed to a stage when the mass is no longer plastic, the crystals have begun to interlock and the initial set has taken place. It is possible, however, to overwork, or "kill," this set, as it is commonly called. If the mixing be continued through this period, the union of the crystals that had just begun is broken up, and it may be some time before a secondary initial set manifests itself. A cement that has had the energy of crystallization once checked in this manner does not yield as high test results as it otherwise would. This overworking of a cement in the tester's hands is analogous to the addition of water and reworking (retempering) a concrete mixture in the actual work, which is a practice strongly condemned.

8. The first of the initial sets might be called the "flash" set, and can easily be recognized by a careful manipulator. The "killing" of a set, of course, always takes place in the operator's hands, but on several occasions another form of secondary initial set has been observed in which, after being in the mold 20 minutes or more, an initial set had apparently taken place; but upon examining in an hour, the mass was found to be plastic again, and the initial set would not follow for perhaps an hour more. It is in view of these facts that the recommendation is made to work quick-setting cements about 1 minute and slow-setting cements 3 minutes, thereby introducing uniformity into the method of testing, that different operators may be enabled to obtain similar results.

9. Effect of Temperature on Time of Setting.—It has already been stated that in making tests the temperature of both water and cement should be the same and that about 60° or 70° F. This is very necessary from the fact that it exerts a very great influence on the time of setting, a high temperature accelerating and a low temperature retarding the set. No definite measure of retardation can be laid down, as it varies with the composition of the cement, its fineness, and age, and whether kept in a moist or a dry place. In some cases, we have observed that samples which took their initial set in 3 minutes at 80° F. required 2 hours at 60° F. and 8 hours at 40° F. These may be considered as extreme cases, but observations seem to indicate that where high temperature accelerates setting, it is so very pronounced that a difference of only a few degrees would change a cement from a class to be condemned to that of a very acceptable cement. Again, the other conditions affecting the set may be such, and, in fact, usually are, if the cement is properly made, that a change in temperature of 10° F. would have no other effect than a change of a few minutes to half an hour in the initial set, thus making the temperature consideration of very little value as far as judging the value of the cement is concerned; but in a cement where other conditions accelerating and retarding are not so well balanced, the temperature has frequently been the final point in deciding the acceptance or rejection of shipments.

10. The foregoing observations were made on American rotary-kiln cements, and, as stated, the effect may be very decided or very slight. Table I, taken from Butler, shows a more uniform effect on vertical-kiln cements and seems to indicate that if other conditions could be held the same, a value could be placed upon each degree rise in temperature.

TABLE I

INFLUENCE OF HEAT ON SETTING TIME OF PORTLAND CEMENT

Sample	Temperature, Fahrenheit							
	100°	80°	60°	40°	100°	80°	60°	40°
	Initial Set in Minutes				Hard Set in Hours			
1	1½	4	6	13	1¼	1½	2	2½
2	3	5	6	8	1	1¼	1¾	2½
3	4	10	15	20	½	¾	1½	6½
4	5	9	15	30	½	¾	1	6
5	6	10	14	25	1	1½	2	2½
6	7	12	15	20	1¾	2	2¼	2½
7	9	10	15	17	3½	6	7	12
8	10	15	35	40	¾	1	1¼	1¾
9	11	15	20	57	3	5	6	10
10	11	13	15	30	2½	3	3½	6
11	19	32	60	120	3	6	7	15
12	15	35	70	360	3½	6	7	22

11. Tensile Strength.—Tensile strength is a measure of the cohesive power when cement is mixed neat (that is, cement and water only) and of the cohesive and adhesive power when cement, sand, and water are mixed. The test is made by molding a neat cement or a sand mixture into *briquets* of which the section at the point of fracture is known. This is sometimes 5 square centimeters (.775 sq. in.), or in this country, more commonly, 1 square inch. Fig. 1 (*a*), (*b*), and (*c*) shows the various shapes of briquet molds. They are made in two parts; when in use, the parts are held together by springs, Fig. 1 (*d*), or by clamps, Fig. 1 (*e*).

12. In making briquets for testing purposes, the first thing to do is to determine the amount of water necessary to get the cement to the proper consistency for filling the molds. Theoretically, not more than 8 to 10 per cent. of water is necessary to complete the chemical reactions involved in setting or hardening, but, practically, about double this amount is required to bring about the desired result. The excess does not take part in the reactions, but merely serves as a medium for bringing atoms in contact, to permit the chemical affinity of the constituents to exert itself. During and after hardening the excess of water evaporates. If an attempt is made to work up a cement neat with 10 per cent. of water, it will be found to work very dry

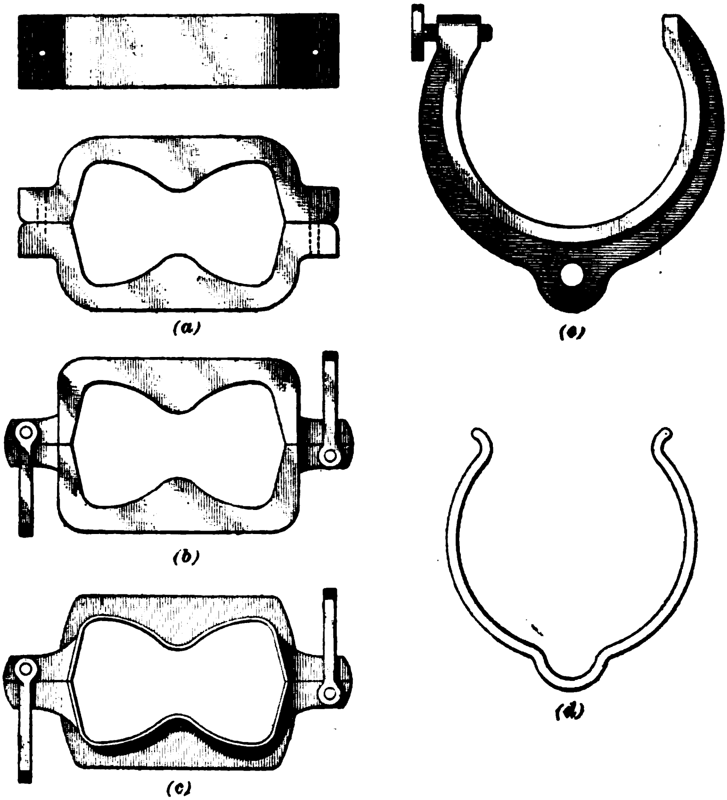

FIG. 1

and the mechanical difficulty in bringing all particles into contact with the water cannot be overcome. Little particles will set, enclosing in them small masses of dry cement, and the reaction will never be complete. Even were it possible to mix thoroughly, the mass would be so dry as to present serious difficulty in molding and, moreover, evaporation during hardening would leave even less water than theoretically required.

As stated above, it is impossible to effect reactions with just the amount of water needed, and the excess serves a double purpose of acting as a medium to facilitate contact for chemical reasons and to make the mass plastic for molding from a mechanical point of view. The amount of water required in practice varies from about 18 to 22 per cent., and this is determined by adding the water slowly and mixing with trowel, spatula, or hand until the mass becomes plastic, and on beating into a small cake water just rises to the surface. The amount being determined, all subsequent briquets of the same sample are made up accordingly. Where a great number of samples of the same brand are to be tested, the operator determines the amount of water necessary and works all up on the same basis, observing, however, at the time of mixing whether any require more or could have been worked satisfactorily with less.

13. The gauging of cement is one of the most important factors in determining the results of the tests. If too little water be used, the mass works up dry, as stated, and a smooth surface cannot be obtained on the briquet; the result is that low tests always follow. On the contrary, if too much water be used, the mass becomes almost liquid and there is a greater amount of water to be removed by evaporation; in this case the briquet shrinks and low tests follow.

The amount of water having been determined, it is necessary to observe uniformity in method and time of working. Specifications as to details are numerous, but it may be well to state that, in general, the time of working should not be less than 1 minute for quick-setting cements nor longer than 3 minutes for slow-setting cements. The tensile strength that a cement develops depends much on the skill in making up briquets, and different testers often obtain widely different results. To obtain uniform results, one must observe the same details in each test. All authorities agree that both the cement and water should be about the same temperature, which should be between 60° and 80° F., preferably 65° to 75° F. A low temperature retards the setting and a high temperature accelerates it. After the cement is properly worked, it is pressed into all corners of a greased mold with a trowel point or the fingers and packed solidly without tamping. When the mold is filled, it is struck off level with the trowel and the cement permitted to harden. During the time of setting it is well to cover with a damp cloth, and, if desired, the initial and final sets may be observed here. The briquets may be taken from the molds after the final set, or after 12 hours, if so specified. It is better, however, to remove the briquets from the mold as soon as sufficiently hard.

14. Testing Briquets in Series.—In testing a cement, it is customary to make up a number of briquets from the same sample and treat them as follows: From two to six are put in water after the final set and broken at the end of 24 hours. This is the 24-hour water test. The others are left in air 24 hours, when a similar number are broken for the 24-hour air test and the remainder placed in a tank of water and preferably set on edge. At the end of 7 days, 1 day in air and 6 in water, the 7-day test is made on a like number. Tests are also made at the end of 28 days, 3 months, 6 months, and 1 year.

15. Sand and Cement Briquets.—These are made up in much the same way as neat, except that standard sharp, quartz sand is used, of such a fineness that it will pass a 20-mesh and be caught on a 30-mesh sieve. The proportions may be two of sand to one of cement, three to one or even five to one, and briquets made the same as for neat tests except that less water is used, a three-to-one mixture, for instance, requiring only about 10 per cent. of water. The cement and sand should be thoroughly mixed dry before adding the water.

16. Breaking Briquets.—The breaking of briquets is accomplished by placing the briquet in a clip and applying a tension uniformly until fracture takes place. For this purpose, two styles of machines are in common use,

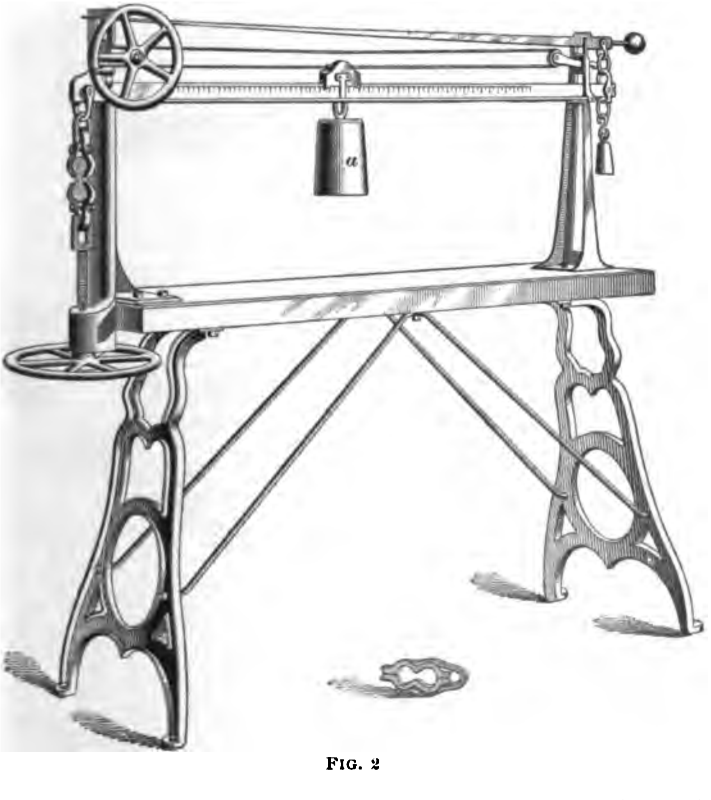

FIG. 2

the **Riehle** and the **Fairbanks**. In the former, shown in Fig. 2, the tension is gradually applied by running a weight *a* out on the scale beam, either by a hand-feeding device or more regularly by machinery. This machine requires the constant attention of the operator, while the Fairbanks, shown in Fig. 3, depends for its tension on the increasing weight produced by a stream of shot from the hopper *c* pouring into a bucket *a* suspended from the end of the scale beam *b*, and when a sufficient weight of shot has fallen in, the briquet is broken and the scale beam falls and automatically shuts off the supply of shot. The broken parts are taken from the clips and the bucket of shot suspended at the opposite end of the scale from the hook *d*, the weights suspended from the hook *e*, and, by means of weights on the pan and a sliding weight *f* on the beam, the number of pounds required to fracture may be read off.

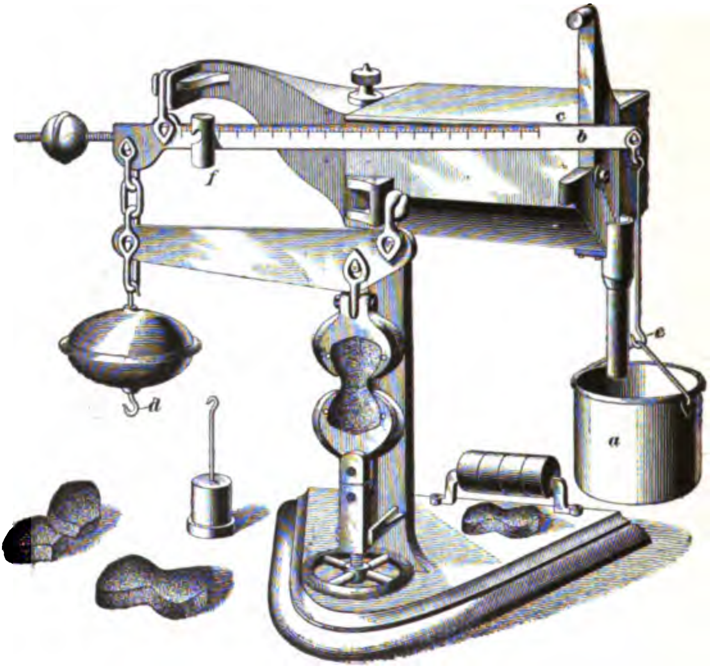

FIG. 3

To obtain uniform results, even in samples of the same cement, it is necessary in making briquets to observe all the precautions given in the following paragraphs. With briquets made under the same conditions, one briquet in a set may break 20 to 30 per cent. lower than the rest by failure to observe proper precaution in placing it in the clip on the machine. It must fit the clip perfectly and must be put in true and square, so that the strain may be vertical and tensional without any torsion. Defective briquets or those made under different conditions should not be broken with any intention of comparing results, as they give figures upon which it is unsafe to judge the cement.

17. Effect of the Speed at Which Strain Is Applied. This affects very largely the results obtained in breaking briquets and is a point too often neglected. Without a uniform rate agreed on, different testers, observing as nearly as possible all other precautions, may get results differing 25 per cent. Faija and Butler made a number of experiments, the results of which were published in the Proceedings of the Institution of Civil Engineers for 1883. From Table II, it will be observed that they found that the tensile strength increased as the speed of applying the strain was increased.

TABLE II

RELATION OF SPEED OF BREAKING TO TENSILE STRENGTH OF BRIQUETS

Number of Briquets	Speed. Pounds in Seconds	Average Tensile Strength
129	100 in 1	560.75
129	100 in 15	506.43
145	100 in 15	452.20
145	100 in 30	430.96
90	100 in 30	417.27
90	100 in 60	403.05
40	100 in 60	416.75
40	100 in 120	400.87

Beginning at the slowest speed of 100 pounds in 120 seconds, they found the increase to be as follows:

3.96 per cent. in 60 seconds.
7.48 per cent. in 30 seconds.
12.41 per cent. in 15 seconds.
23.14 per cent. in 1 second.

They finally adopted a rate of 100 pounds in 15 seconds, which is now generally used.

18. Points to Be Observed in Testing Cement.—As the quality of a cement is judged almost entirely from its physical tests, great attention being paid to tensile strength, and, as stated before, widely differing results are obtained by different testers and even by the same tester on the same sample in spite of all good intentions to follow the same details, it may be well to recapitulate some of the points made by John Newman in his book on concrete.

1. The nature of the plate upon which briquets are made, whether impervious or porous.

2. Kind of water. It should be spring, cistern, or river water, but neither distilled nor boiled.

3. Temperature of water and cement.

4. Amount of water.

5. The mold should be perfectly clean and the inner surface oiled slightly, to prevent the cement from adhering.

6. Area of breaking section.

7. Length of time in mold after setting.

8. The position of strain with regard to the breaking section of the briquet.

9. The nature of strain, whether slowly or rapidly applied.

10. The form of clip in the breaking machine.

11. Whether clip is hung on pivots, to prevent cross strain.

12. The equal or unequal bearing of clips upon briquets.

13. The age of cement after grinding.

14. Whether the cement has been properly air slaked.

15. Whether briquets are made by the same operator on the same day and under the same conditions.
16. Length of time required in gauging or mixing.
17. Method of filling the mold.
18. Whether the mold is packed by hand or machine and whether tamped or only pressed by the fingers and trowel.
19. Whether briquets are covered by damp cloth during setting period.
20. Method of gauging dry cement.
21. Fineness of the dry cement.
22. Season of year.
23. Skill in handling of testing machine.

19. Tensile Strength of Various Brands of Cement. The following figures (Table III) represent the average of a number of tests made upon various cements, but it must not be inferred that the same results would be obtained at the present time, as a deviation from any of the foregoing conditions might alter the figures considerably and, moreover, no brand of cement runs absolutely uniform in results even in the same hands.

TABLE III

TENSILE STRENGTH IN POUNDS PER SQUARE INCH

Name of Cement	Neat					One Part Cement, Three Parts Standard Quartz Sand			
	24 Hr.	7 Da.	28 Da.	3 Mo.	6 Mo.	24 Hr.	7 Da.	28 Da.	6 Mo.
Alpha	294	660	780	820	887	52	194	269	311
Atlas	440	638	728	755	802	94	189	242	348
Giant	305	452	524	598	698	90	161	227	332
Hemmour	221	466	502	573		84	191	263	
Lehigh	281	659	748	777	790	63	214	297	
Mannheimer	211	483	585	660		74	177	237	
Vulcanite	263	724	794	836	885	71	225	279	359

20. Specifications for Tensile Strength.—These vary, depending somewhat on the work for which the cement is intended, but more on the views of the engineer in charge of the work on which it is to be used. As a rule, the minimum for a 24-hour neat test is placed between 125 and 250 pounds per square inch. It is seldom that a higher test than 250 pounds is called for as a minimum, and if any specifications are made, less than 125 pounds is never given as a minimum. For the 7-day neat test, any figure between 350 and 600 pounds may be placed as a minimum. On sand briquets, 3 parts of sand to 1 of cement, no requirement is made for the 24-hour test and the 7-day minimum is usually placed at 125 pounds. It must be borne in mind, however, that the long-time tests are also taken into consideration, but in no case is a cement held for longer than the 28-day test, the longer-time tests being based on the previous reputation of the brand.

21. Comparisons of Tensile-Strength Tests.—It is also to be noted that cements which develop the greatest strength in 24 hours and 7 days, and even including 28 days, are not necessarily the highest quality. For instance, one may run 500 pounds in 24 hours and 800 pounds in 7 days but fall back to 750 pounds in 28 days, or it may run 350 pounds in 24 hours, 650 pounds in 7 days, 900 pounds in 28 days, and fall back to 800 pounds in 3 months. Such are called *retrograding cements* and are inferior to those ranging from 250 pounds in 24 hours to 600 pounds in 7 days, 750 pounds in 28 days, and 850 pounds in 3 months. A good cement should increase in strength at a gradually diminishing rate for an almost indefinite length of time, and although it rarely increases after 2 years, the long-time tests should show no falling off. Such cements are called *normal* and are the ones to be desired. A common mistake is made in judging cements on short-time tests if high strengths are considered, as such tests are very likely to prove retrograding.

22. Compressive Strength.—Tests showing the resistance to compressive strain indicate more nearly the actual conditions to which a cement is generally subjected in practical work, but the difficulty in obtaining uniform results has stood in the way of adopting it. In Germany, some attention is given to it; in England, it is discouraged; and in the United States, a number of laboratories are now engaged in making them in a spirit of research with a view to incorporating the specifications into contract work, but at present tensile strain is used largely to judge the quality. In practical work, a combination of both tensile and compressive strains is more commonly the case, and tensile tests, being more uniform, are universally adopted.

In making compressive tests, cubes of various sizes are used, molded in the same way as for tensile strain, and, of course, mixed and worked in a similar manner. The apparatus for testing must be of entirely different design and much stronger, as the crushing strain is from six to ten times the tensile strain. The principal difficulty is in distributing the pressure evenly over the surface of the cube.

23. Constancy of Volume.—This is determined by making pat tests. Cement is mixed as for neat briquets except that more water is used, generally from 22 to 27 per cent. After it is thoroughly worked, the mixture is placed upon a glass plate about 4 inches square and worked into a cake or pat, about 3 inches in diameter, $\frac{1}{2}$ inch thick at the center, and drawn out to a thin edge on the circumference, care being taken that the cement adheres firmly to the glass, leaving no air bubbles between. Instead of a round pat, it may be worked in a wedge about 2 by 3 inches, $\frac{1}{2}$ inch thick at the base and drawn to a point. The following tests may be made.

24. Normal Pat Test.—Two pats are prepared as described and as soon as made are placed in a damp atmosphere—a zinc-lined drawer with water under a false bottom is convenient, but any vessel may be used—to prevent drying on the surface. After 24 hours, one pat is placed in water and the other under cover and their condition reported at the end of 7 days and 28 days, although if the pat stands the 7-day test, the latter is usually satisfactory. If at the end of that time the pat is sound and hard and still adheres to the glass, it passes; if, on the contrary, it swells, cracks, warps, or leaves the glass, it fails on this test. The expanding, cracking, or disintegrating of a cement is frequently called *blowing* or *checking*.

25. Hot-Air Test.—A pat is prepared on moist filter paper and set on a glass plate. After setting the paper is removed and it is placed, as before, under cover for 24 hours; it is then placed in an air bath at a temperature of 212° F. and its condition noted, as before. If it swells, cracks, or warps, it fails to pass.

26. Warm-Water Test (Falja's).—The pat is prepared as above and immediately placed under cover over a water bath having a temperature of 115° F. After the final set it is lowered into water and kept at 115° F. for the remainder of the 24 hours and its condition noted as before.

27. Boiling-Water Test (Michaelis').—A pat is prepared on filter paper or glass and treated as in the hot-air test, and at the end of 24 hours it is placed in a bath of cold water. This is then heated to boiling and kept there for 3 hours, when its condition is noted.

In any of the preceding pat tests a warping, swelling, or cracking indicates a lack of constancy in volume, and the cement is considered defective. The checking, as it is called, may be due to any one of the following causes: (1) Excess of lime; (2) deficiency of lime; (3) imperfect preparation of raw material; (4) underburning; (5) presence of sulphides; (6) presence of too much sulphate of lime; (7) presence of too much magnesia; (8) coarse grinding of finished product.

28. Unsoundness Due to Excess of Lime.—It is said that the amount of lime permissible in a cement without injuring its soundness is steadily increasing, but it by no means follows that all cements can be run as high as 66 or even 64 per cent. of lime. Where the raw material has been thoroughly ground and mixed and the burning apparently all that could be desired, a 64-per-cent. lime cement may check, while with the same materials, but run on a 63-per-cent. lime mixture it may stand all tests. The maximum amount of lime is overstepped when, with proper mixing and pulverizing of raw material, thorough burning and fine grinding of finished product, it yields a cement defective in pat tests. The tendency to produce cements high in lime is held in check by increased cost of burning and danger of failing in tests.

29. Unsoundness Due to Deficiency of Lime.—This is a defect that seldom manifests itself, as a cement may run rather low in lime without checking and before the low limit in lime, causing checking, is reached it has passed the low limit causing quick setting, and the chemist, to avoid too slow setting, necessarily keeps the lime high enough to prevent checking. In cases where checking is traceable to low lime, the defect usually becomes apparent in a short time, whereas with high lime it may take several months. This latter applies to cement in practical work. In laboratory tests it shows up at once.

30. Unsoundness Due to Imperfect Preparation of Raw Materials.—In case the raw materials are not ground sufficiently fine or mixed intimately enough before being brought to the kiln, we have in the latter the formation of clinkers, showing a lack of uniformity, and in different samples or different parts of the same sample we may find spots showing a fusible low-lime mixture and in others a high-lime effect, and when these are ground the combined effect of high- and low-lime mixtures is obtained.

31. Unsoundness Due to Underburning.—A clinker that has not been sufficiently burned is soft, porous, and easily ground, and if exposed to the atmosphere for a short time, the free lime rapidly becomes hydrated and sound pats are obtained. Any defect due to underburning usually manifests itself in a very few days, at the end of which time the lime is fully hydrated and further deterioration does not take place; but in case of overlimed cements, it may be some weeks before cracking becomes evident and they may in time disintegrate entirely.

32. Unsoundness Due to Sulphides.—This is of rare occurrence and is due to the oxidation of the sulphides to sulphates, causing checking and cracking. In case of sulphide of iron, it is very marked, as it first passes to protosulphate and afterwards to the oxide or oxides.

33. Unsoundness Due to too Much Sulphate of Calcium.—The addition of 1 to 3 per cent. of gypsum or plaster of Paris to a cement has the effect of retarding the time of setting considerably, owing to its preventing the rapid hydration of aluminate of calcium by forming sulphoaluminate of calcium. The combination of sulphate with the aluminate of calcium takes place slowly. If an excess of sulphate is added to the cement, this reaction does not take place until some time after the hardening of the silicate and causes a swelling or cracking by taking up water in crystallization. In silicate-of-calcium cements, where alumina is absent, the sulphate of calcium has no other effect than to impoverish, or weaken, the concrete by gradually dissolving, but does not produce swelling or cracking, since there is no alumina there to form the sulphoaluminate of calcium.

34. Unsoundness Due to too Much Magnesia.—The presence of too great an amount of magnesia has very much the same effect as free lime. It causes the cement to expand excessively and crack. The effect of magnesia in Portland cement is more fully discussed later.

35. Unsoundness Due to Coarse Grinding.—A very common cause of checking in pat tests is coarseness in raw material. During the past few years, with the improved pulverizing machinery, the raw material is fed into the kilns in a finer state of division and in a more intimate mixture than formerly, and it has been found possible to steadily increase the percentage of lime, which, some years ago, ran from 56 to 60, then from 60 to 62, and now many run 63 to

64 and even to 66 per cent. While it is not necessarily a rule, the cements highest in lime have up to the present been burned in vertical kilns and, therefore, at a lower temperature and during a longer period.

Fine grinding of the burned cement is also necessary when high in lime, as the free lime in the coarser particles does not become aerated or slaked as in the finely divided cement, and in case of coarse cement, it may be in the work for months before any unsoundness develops.

36. Specific Gravity.—The **specific gravity** of cements is a point that has never received much attention either from manufacturers or inspectors. For some years attempts were made to compare different brands by their weight per bushel or per cubic foot. The fallacies of this become evident when the difficulties in devising a method of packing the cement uniformly are considered. Several have been proposed and used to some extent, but the variations, even on the same sample and with the same operator, are such as to render tests valueless. In one attempt to determine weight per cubic foot, results ran from 90 to 110 pounds, depending on whether the cement was loosely thrown in and then struck off level or whether shaken down, repeatedly filled up again, and struck off level. Of course, with a machine to pack it, the difference is not so great, but nevertheless it is sufficient to make useless any attempt to judge a cement by weight per bushel or per cubic foot. The weight of a cement varies with (1) thoroughness in burning; (2) fineness of grinding; and (3) age.

37. A cement that is properly burned usually has a rather high specific gravity, while with one that has been underburned it is usually low. All other conditions being the same, an exact determination of the specific gravity would be an index to the burning. In making a comparison of this nature, the age of cement must be taken into consideration. A fresh sample shows the highest specific gravity. If exposed for any length of time, cement absorbs moisture and carbon dioxide from the atmosphere, and as both are lighter than cement its density diminishes. Faija observed in one case a loss in weight per bushel of 11.5 per cent. in 6 months and 15.9 per cent. in 1 year. On a number of other samples he observed, by exact specific-gravity tests, a loss of 4 to 8 per cent. in 6 months. In determining weight per bushel, burning, grinding, and age have their effects, coarse grinding making a heavier cement, while in the specific-gravity test, only burning and age affect the result, provided the chemical composition is uniform in various samples. As there is such a slight variation in composition, this may be neglected in comparing samples from any particular mill, and an index to the burning is obtained if the samples are of the same age and exposed under the same conditions. The specific gravity of a well-burned cement varies from 3.10 to 3.15; in poorly burned cement, it may run less and in overburned a trifle higher.

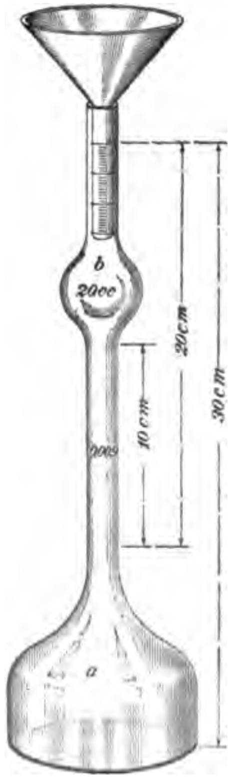

FIG. 4

38. Le Chatelier Specific-Gravity Flask.—Several styles of apparatus for the determination of specific gravity have been devised, but as the value of the test is open to question, only one, that of Le Chatelier, will be described. As will be seen in Fig. 4, it consists of a flask *a* of about 120 cubic centimeters capacity, with a neck about 9 millimeters in diameter and 20 centimeters long. Half way up the neck is a bulb *b* of exactly 20 cubic centimeters capacity, as indicated by the marks above and below. Beginning at the upper mark, the tube is graduated in tenths of a cubic centimeter from zero to 3 cubic centimeters.

As cement has hydraulic properties, it is evident that in using this or any similar apparatus a liquid having no effect on the cement should be employed. Petroleum, turpentine, or benzine may be used. The latter, while very volatile, gives good results if proper precautions are used.

39. Determination of Specific Gravity With Le Chatelier's Flask.—Fill the flask to the bottom mark on the neck, and having weighed out 64 grams of cement, begin to introduce it through a funnel whose stem reaches just below the upper graduations, so that no cement sticks to the side above this point. Cement is added until the liquid reaches the zero mark on the neck, and the remainder weighed, when by difference the weight of cement (equivalent in volume to 20 cubic centimeters of water) is obtained. Another method is to introduce all the cement and read the number of cubic centimeters on the scale, which, added to the 20 in the bulb, gives the volume of liquid equivalent of 64 grams, whence the specific gravity is easily calculated. In making a test, care should be used that no air bubbles be permitted to remain in either the flask or tube, and those carried down by the cement should be removed by gentle tapping. It is quite essential that the temperature should be the same at the end as at the beginning of the operation, and it is advisable, especially when benzine is used, to keep the flask submerged in cold water during entire work.

CHEMICAL ANALYSIS OF PORTLAND CEMENT AND RAW MATERIALS

ANALYSIS OF PORTLAND CEMENT

NOTE.—In connection with these analyses, the student is referred to *Quantitative Analysis*, taking especial note of the precautions given.

40. Introductory.—The principles involved in quantitative analysis of cement differ in no way from those laid down for other mineral analyses, but specialization being necessary in all lines of work, it is natural that cement chemists should evolve methods best adapted to the material in hand.

Cement occupies an intermediate place between feldspars and clays, containing insoluble silicates and limestones and carbonates soluble in acids. In the former, it is necessary to fuse the substance with carbonate of soda to break up insoluble silicates, while in the latter, unless there is considerable silicious matter present, a simple treatment with dilute hydrochloric acid will effect solution. In cement laboratories, it is necessary to arrive at fairly accurate results in a short space of time, and any method yielding results accurate to .2 per cent. will serve the purpose, as two samples from the same barrel and analyzed by the standard and more accurate methods will be found to vary that much.

Portland cement will be found to dissolve more or less completely on digestion with dilute hydrochloric acid; but if the residue be fused with sodium carbonate, it will be found to contain small amounts of iron, alumina, and lime. If the following method is followed, these amounts will be so trifling as to make the sacrifice of accuracy incommensurate with the saving in time.

41. Determination of Silica.—One-half gram of finely pulverized and dried cement is placed in a 4-inch evaporating dish and to it is added 10 cubic centimeters of hydrochloric acid, specific gravity 1.20, diluted with an equal volume of water, and a few drops of concentrated nitric acid added. The dish is covered with a watch glass and placed upon a sand bath, where the acid will gently boil away in a couple of hours. In order to effect the complete breaking down of soluble silicates and the dehydration of the silica, it is necessary to allow it to remain on the water bath until no odor of acid is given off. A little heating over a free flame, to drive off the last traces of acid, will do no harm if carefully done and the heat is not too high. Allow the dish and contents to cool and add 20 cubic centimeters of dilute hydrochloric acid (1 part of strong acid to 1 of water), then cover and boil gently for a few minutes. Add 50 to 100 cubic centimeters of water and boil briskly. Allow to settle and filter, washing afterwards with hot water until the filtrate shows no opalescence in the solution of nitrate of silver. Dry the residue, place in a weighed platinum crucible, and ignite until white, then cool, and weigh as silica SiO_2. The weight of the SiO_2 multiplied by 100 and divided by the weight of substance taken gives the percentage in the cement.

42. Determination of Iron and Aluminum Oxides. The filtrate from the silica is heated gently, a very slight excess of ammonia added and then boiled for a few minutes. Allow this to stand until the precipitate has settled and filter, washing several times with hot water; then with a jet of hot water wash the precipitates back into the same beaker and dissolve in a little dilute hydrochloric acid. Add water enough to bring the volume up to 100 cubic centimeters and reprecipitate with ammonia. Filter upon the same paper, wash thoroughly with hot water, dry, ignite, and weigh as oxides of iron and alumina $Fe_2O_3 + Al_2O_3$; then calculate the percentage in the usual manner. The reprecipitation is necessary, as in the presence of a large amount of lime, iron and alumina always carry some of the lime down with them.

It is rarely necessary to separate iron and alumina, but if desired, 1 gram of the sample may be taken at the start and the filtrate from the silica divided, and iron and alumina precipitated in each half separately. One precipitate is treated as above and the other is washed from filter paper into a small Erlenmeyer flask; the funnel is then placed in the flask and the remaining precipitate dissolved and washed into the flask with a little hot, dilute sulphuric acid. The volume should be about 200 cubic centimeters. To this add 1 gram of finely pulverized zinc in which iron has been previously determined and put the stopper with a rubber valve into the flask. When the zinc is completely dissolved, titrate the iron with a weak standard solution of potassium permanganate. Calculate to iron oxide Fe_2O_3 and subtract from the double oxides for alumina. The difference will be the weight of the alumina in $\frac{1}{2}$ gram of cement. The percentages of Fe_2O_3 and Al_2O_3 are calculated by multiplying their weights by 100 and dividing the results by the weight of the sample taken.

43. Determination of Lime.—The filtrate from the iron and alumina, which by this time is about 300 cubic centimeters in volume, is made slightly more alkaline by the addition of a few drops of ammonia and brought to boiling. At the same time, about 20 cubic centimeters of a saturated solution of ammonium oxalate is heated to boiling in a separate beaker. This is then added to the filtrate and kept boiling for several minutes; it is then set aside to cool and settle. By precipitating in above manner, oxalate of lime settles rapidly in crystals, permitting of rapid filtering and washing. The filter should be washed repeatedly with hot water to insure removal of the last traces of ammonium oxalate.

The precipitate is then washed from the funnel into the beaker and the filter paper washed several times with hot dilute sulphuric acid, to remove the last of the calcium oxalate. The solution is now made up to 300 cubic centimeters and about 10 cubic centimeters of strong sulphuric acid added; then the whole is brought almost to the boiling point and titrated with a standardized solution of potassium permanganate, as directed in *Quantitative Analysis.*

44. Determination of Magnesia.—Magnesia is determined in the filtrate from the calcium oxalate. This is cooled as much as possible; then 20 cubic centimeters of a saturated solution of ammonium phosphate is added and the whole stirred frequently. Allow it to stand in a cool place over night, filter, wash, ignite, and weigh the magnesia as pyrophosphate $Mg_2P_2O_7$. The weight of this multiplied by 36.04×100 and divided by the weight of the sample taken gives the percentage of MgO in the cement.

45. Determination of Sulphur.—Three to 5 grams of the sample is digested with hydrochloric and a little nitric acid and evaporated to dryness as for silica, taken up with dilute hydrochloric acid and the silica removed as described, except that as little hydrochloric acid as possible should be used to take up the soluble portion. The filtrate is boiled and 15 cubic centimeters of a boiling, saturated solution of barium chloride added and allowed to stand. When the precipitate of barium sulphate has settled, it is filtered, washed with hot water, dried, ignited, and weighed, and sulphuric acid calculated from the barium sulphate by multiplying the weight of the $BaSO_4$ by .34335 × 100 and dividing by the weight of the sample taken. The percentage of SO_3 in the cement is found by multiplying the weight of the SO_3 by 100 and dividing by the weight of the sample taken.

ANALYSIS OF RAW MATERIALS

46. Introductory.—It is scarcely necessary to state that a careful examination of all raw materials used is of the utmost importance. In fact, it is of more importance than the analysis of the finished cement, for if the raw material is properly mixed and the subsequent steps in the process are carefully carried out, the chemical composition of the resulting cement cannot be other than that desired. On the other hand, if the proportions of raw material are not correctly determined, no subsequent step can eliminate this error and a poor cement results. The chemist's duties, therefore, begin in the quarries, and he should have complete analyses of all the cement rock, limestone, clay, or other raw materials that it is proposed to use.

The methods used do not differ in any respect from the standard analytical methods for limestones and clays. Having complete chemical data as to ingredients to be used, the proper proportions are then calculated and the mixture made. In most mills, the chemist has from 1 day to 1 week in which to get analyses of raw materials before they are sent through the mill. This enables him to determine carefully what the mixture should be. If the material runs very uniform throughout, the proportions can be regulated in this way, but usually there is so much variation in the material that it is necessary to check its composition as it goes to the kiln at least several times a day. Time is the great factor here, and it is essential that a method should be used that will give approximate results on lime or calcium carbonate in a very few minutes. The following are quick methods:

47. Determination of Calcium Carbonate. — The determination of calcium carbonate may be made by either of two methods. First, by acidimetry, and, second, by measurement of the CO_2 evolved on the treatment with an acid.

First Method.—This is a simple, easy method, requiring little apparatus and giving fairly accurate results. It is applicable, however, only where the carbonate is the only constituent soluble in a weak acid solution. In any other case, it can only be discreetly used by the chemist if he has carefully prepared a table of corrections applying to the materials in hand. Its principle is that of decomposing calcium carbonate by a standard acid solution and then titrating the excess of acid. A convenient strength of acid is one of which 1 cubic centimeter equals about .02 gram calcium carbonate. A standard alkali, either caustic potash or caustic soda, is made up so that 1 cubic centimeter is equivalent to 1 cubic centimeter of the acid. This is not necessary, but facilitates calculation. The acid may be standardized against pure calcium carbonate, or, what is perhaps better, determine lime gravimetrically in a sample of mixed raw material and use this for standardizing the acid.

Having determined a standard for the acid, say 1 cubic centimeter = .02140 gram $CaCO_3$, the method is as follows: Weigh out one gram of mixed material and transfer it to a convenient sized Erlenmeyer flask, add about 50 cubic centimeters of distilled water, and then add 40 to 45 cubic centimeters of the standard acid solution. Close the flask with a rubber stopper through which a glass tube about 2 feet long is placed to serve as a condenser and boil for a few minutes or until effervescence ceases. Add a few drops of phenol-phthalein and titrate the excess of acid. By difference, the acid necessary to decompose carbonate is known.

EXAMPLE.—One gram of the mixture was boiled with 45.4 cubic centimeters of standard acid, and 10.2 cubic centimeters of alkali was required to neutralize the excess of acid; therefore, 35.2 cubic centimeters of acid was required to decompose the carbonate of lime. One cubic centimeter of standard acid = .0214 gram of $CaCO_3$. .0214 × 35.2 = .75328 gram, or 75.32 per cent. of calcium carbonate.

Second Method.—The method of measuring the carbon dioxide liberated requires more apparatus, careful attention to details of manipulation, correction for temperature and barometric pressure, and, in the end, gives results no more accurate, practically speaking, than the foregoing method if a proper table of corrections be prepared. The method is used in some laboratories, however, and therefore deserves some attention.

The apparatus used is termed a **calcimeter** and may be of various designs. The first devised for this purpose was that of Dr. Scheibler, but has been replaced by the more modern apparatus of Dietrich, or the still later modified form of Faija. The following figures show the two forms of apparatus, of which only the application of the Faija will be described, the Dietrich then becoming self-evident.

48. Faija's Apparatus.—This apparatus, shown in Fig. 5, consists of the following parts: a bottle *a*, for decomposing carbonate, thus generating carbon dioxide; an acid measure *b*, of 10 cubic centimeters capacity; a glass or gutta-percha tube *c*, for introducing acid; a condenser *d*, with lead or glass coil; a tube *e*, for measuring the gas; a stop-cock *f*, for preventing return of gas to condenser; an outlet stop-cock *g*; an equilibrium tube *h*, adjustable in any vertical position on rod *i* by means of thumbscrew *k*; a barometer *l*, for adjusting pressure; a cock *m*, for closing tube between barometer and equilibrium tube; an india-rubber ball *n*, which may be compressed or expanded by handle *o*, to regulate pressure in barometer.

The apparatus is set up perfectly vertical and firmly fastened. The tube *h* is lowered until its top is on the same level with lower reading on tube *e*. The upper rubber tube is then removed and a funnel inserted and the tube *h* filled with distilled water, care being taken that no air remains in rubber tube connecting *h* and *e*. While filling, cocks *f* and *g* should be open. The other rubber tubes are now connected as shown in the figure, and, in order to determine whether they are perfectly tight, the tube *h* should be raised (the cock *g* being closed) until there is several inches difference in the height of water in *e* and *h*, and allowed to stand for a few minutes. If this difference remains constant, there is no leak.

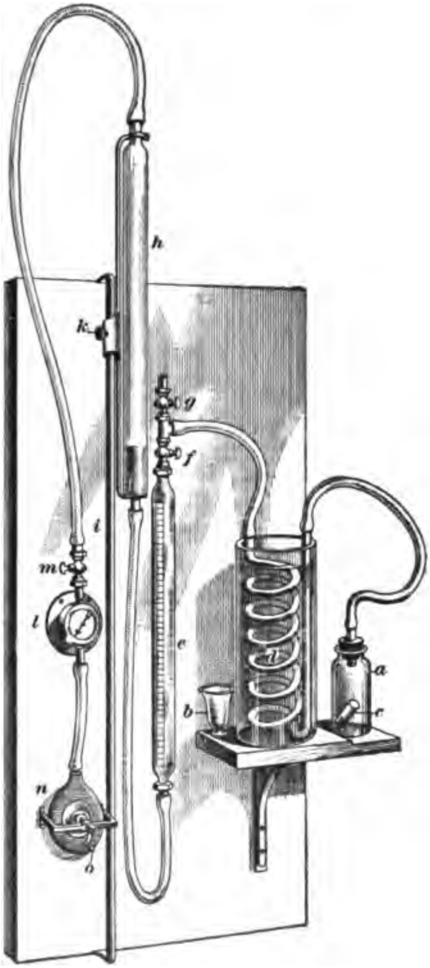

FIG. 5.

The details of manipulation are as follows: Open bottle *a* and cocks *f*, *g*, and *m*. Raise the tube *h* to such a height that the water in *e* is exactly level with the mark under the cock *f* and fasten *h* at this point. Remove the acid measure from *a* and insert in the bottom of *a* the weight of carbonate desired to test. Place the measured amount of acid in *c*, being careful to allow none to remain on the outside, and with tongs insert it open end up in bottle *a*, as shown in the figure. Close *a* carefully, also cock *g*. Slack the thumbscrew *k* and hold *h* in one hand and *a* in the other; then incline the latter until the acid runs out upon the carbonate, and as the gas is generated and driven over into *c* lower *h* slowly, keeping the water at same level in both tubes. Shake the bottle *a* during the operation and be sure all the acid runs out of *b*. If any heat is generated in *a*, thus raising the temperature of the gas, cool *a* in the condenser. Repeat the operation several times until there is no more evidence of action of the acid on the carbonate. This can be determined when the level of the water in both tubes remains constant. Close cocks *f* and *m* and turn handle *o* in one way or the other until the barometer reads normal pressure, that is, 760 millimeters, or 29.92 inches. Bring *h* to a point where the level of the water in *e* and *h* is exactly the same and read the volume from *e*. The

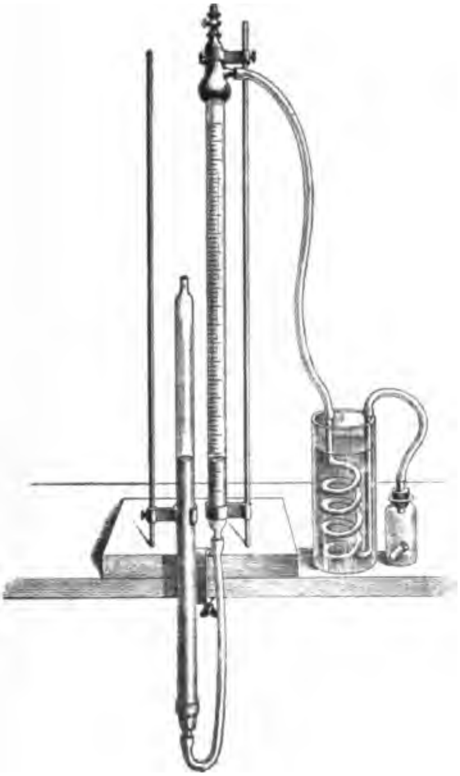

FIG. 6

pressure being normal, the reading need only be corrected for temperature, as directed in *Quantitative Analysis*. The temperature may be taken from the condenser, but this water as well as that in the tubes should be of the same temperature as the room. One cubic centimeter of CO_2 at 0° C. weighs .0019712 gram. The weight of CO_2 multiplied by 2.2743 and by 100 and divided by the weight of the sample taken gives the percentage of $CaCO_3$. The barometer should, of course, be adjusted to normal pressure before beginning the determination.

49. Dietrich Apparatus.—In using the original Dietrich apparatus, shown in Fig. 6, the barometer reading is taken separately and corrections for both temperature and pressure are made as directed in *Quantitative Analysis*.

NOTES ON PORTLAND CEMENT

50. Gypsum.—**Gypsum** is a hydrous sulphate of lime having the formula $CaSO_4,2H_2O$. It occurs in nature in flat, tabular, monoclinic crystals, very soft and showing good cleavage. When pure, its color may be white to colorless. It also occurs in microcrystalline particles, known as *alabaster*, or in fibrous structure as *satin spar*. By simple calculation from the above formula, it will be found to contain about 21 per cent. of water of crystallization. By heating, it begins to lose this water at about 80° C., and at 200° C. all this is driven off. By careful heating at about 125° C., three-fourths of the water is driven off, leaving a composition $2CaSO_4,H_2O$, known as *plaster of Paris*. This when mixed with water soon "sets," or hardens, owing to a recombination with water forming a crystalline hydrous calcium sulphate. This is explained by Le Chatelier as follows: Plaster of Paris $2CaSO_4,H_2O$ is soluble, and on mixing with water some of the plaster is dissolved and immediately combines with more water, forming calcium sulphate with 2 molecules of water, which, being less soluble, crystallizes out, then more of plaster is dissolved and the process repeated. Theoretically only about 18 per cent. of water is necessary to set a plaster, but it is not possible to carry it out practically, and 30 to 50 per cent. is generally used. When mixed with water, it sets in a very few minutes, swelling or expanding as it does so.

51. Use of Gypsum in Portland Cement.—Freshly ground Portland cement, especially that made in a rotary kiln, is always quick setting. This may be overcome by long exposure to the air, permitting the absorption of moisture and hydration of free lime. In cement burned in a shaft kiln at a low temperature and through a longer space of time, the time of *seasoning* the cement, as it is called, is less than cement from a rotary kiln where the temperature is high and the period of clinkering comparatively short. As it is not practical to hold cement for a long time or turn it over from time to time to expose it to the air, a retarder is added either before grinding or when packing for shipment. The purpose of this retarder is to check the setting, which takes place in from a few minutes to a period of half an hour or more. Chlorides and sulphates have this effect, but in the manufacture of cement a sulphate is generally used.

Gypsum in its natural state or as plaster of Paris is therefore added. The latter in itself is quick setting, as is likewise cement, but when these are mixed in proper proportions, they form a slow-setting mixture. The amount necessary varies from ½ per cent. to 3 per cent. of the cement used. The cause of the quick setting of a fresh cement is the rapid hydration of aluminate of calcium, which compound sets almost as quickly as calcined plaster. The action of the plaster in a cement, as explained by Candlot, is due to the formation of a sulphoaluminate of calcium, which prevents the hydration of the aluminate. The effect of plaster or gypsum in amounts up to 2 per cent. has never been found to be injurious, but by making it possible to use less water gives greater density and consequently higher strength.

52. Magnesia.—The effect of magnesia upon Portland cement is a subject that has held the attention of cement chemists for a number of years, and, while investigations are still being carried on, the consensus of opinion seems to be that its presence in large quantities causes cement to expand and crack after long periods. Candlot, in his "Ciments et Chaux Hydrauliques," says: "Magnesia produces the same effect as free lime; in recent years the many accidents occurring in constructions where magnesian cement had been used have drawn attention to this question. Magnesian cements give very good results in tests, and masonry made with these cements presents all desirable solidity during several months and sometimes several years. But at a given time swelling begins to manifest itself and with such intensity that nothing is able to resist it." He states that a rod of magnesian cement 1 meter long was found to elongate 26 millimeters in 3 years.

Dyckerhoff, who perhaps has given the most thorough study to the subject, confirms the above fully. He also states that more than 4 per cent. of magnesia, whether considered as lime in the mixture or whether added to a normal lime, alumina, silica cement, and considering it inert, causes a steady falling off in tensile strength, and when the amount reaches 8 per cent. causes cracking. In explanation of the above, Fremy believes that magnesium compounds become hydrated much more slowly than calcium compounds, and it follows that a concrete made of a cement which is a mixture of silicates and aluminates of calcium and magnesium will, after the set of the calcium compounds has taken place, undergo changes and molecular movements due to hydration of magnesia, which leads to the expansion and cracking. In view of the above, specifications call for less than 4 per cent. of magnesia.

53. Oxides of Iron.—The percentage of these present in a Portland cement varies from 1½ to 4 per cent., consisting chiefly of the lower oxides. These combine with sulphur and form sulphide, which in turn oxidizes to sulphate of iron.

A briquet may be broken and show a decided greenish tint, but on exposure to atmosphere the sulphate oxidizes and the fracture turns a rusty brown color. The discoloration of cements after going into the work is due to the oxidation of iron present. The part that iron oxide plays in the composition of cement or in its setting is rather obscure, but it is supposed to be similar to alumina in assisting combinations of lime and silica. Le Chatelier, however, states that mixtures of oxide of iron and lime on burning yield products that slake in water but have no hydraulic properties. In ordinary cement mixtures the percentage of iron is so small that it is usually reckoned in with the alumina. Cements high in iron and alumina, however, are not suitable for marine work owing to the fact that ferrate and aluminate of calcium are softened by the water, while silicate of calcium remains hard.

54. Carbon Dioxide.—Carbon dioxide in cement may be due either to imperfect burning in the manufacture, or to absorption of the gas from the atmosphere after manufacture. A fresh cement, if well burned, should show less than 1 per cent. carbon dioxide, while, if poorly burned, it may show as high as 3 or 4 per cent. The amount due to absorption from the atmosphere depends on the age and the kind of package in which it is stored. Faija and Butler made some experiments on this point that, while interesting, are not of great importance to the manufacturer, as in this country a cement rarely reaches an age of 3 months before being used and in many cases less than 1 month. They took a fresh sample barrel from the manufacturer and found it contained .25 per cent. of carbon dioxide. Part of the sample they spread out in a thin layer, exposed to the atmosphere, and the remainder they kept in the cask with head in, as in the original package. From time to time they made determinations of carbon dioxide, with the following results. No. 1 was from the cask and No. 2 that exposed to air. The figures represent percentage of CO_2 absorbed.

TABLE IV

TIME EXPOSED TO AIR

Sample	28 Da.	3 Mo.	6 Mo.	1 Yr.	2 Yr.	3 Yr.
No. 1.....	.57	1.08	1.57	2.05	2.90	3.21
No. 2.....	1.82	3.30	4.00	4.90	5.05	5.60

From these figures it becomes evident that the content of carbon dioxide is not much of a guide to the burning except to the maker, who can determine it upon fresh samples. As in a newly ground cement there is very little absorption of moisture, a determination of loss on ignition serves as an indication of the burning.

55. Alkalies.—The amount of alkali in cement is usually so small that it is not considered either in manufacture or in testing. In the Lehigh district, where two rocks are used, the amount may be said to be practically nothing, as it runs less than ¼ per cent. In cements made from clay and limestone, chalk, or marl, the amount of alkali is usually larger, as in many cases the clay contains from 3 to 5 per cent. of potash and soda, which in finished cement would give about 1 to 2 per cent. of alkali. As in the majority of cements the amount is less than 1 per cent., it has not been given much attention and its effect on the hydraulic properties is a question still open to discussion. As the silicates of potassium and sodium are the only soluble silicates in cement, it seems very probable that they would act as a medium to carry silica to free lime, but, of course, being present in such small quantities, their effect is scarcely noticeable. Newberry, in his "Constitution of Hydraulic Cements," takes the opposite view, that is, that soda has no effect, but having made only a limited number of experiments, makes no positive statement. In burning, the presence of alkali reduces the temperature necessary to clinker, but being present in such small amounts, its effect is estimated more from a theoretical point of view than from practical observation.

56. Coloring Matter in Cement.—In occasional cases, an effect in color is desired in the use of cement. These colors are not natural in the cement, but are added to it by the consumer at the time of using. They have the same effect as any other adulterant, that is, they reduce the strength of the cement in every case, with the exception, possibly, of ultramarine, which is said to increase it if used in small quantities. For color effect the following are recommended:

For black, 2 per cent. of excelsior carbon black.
For gray, ½ per cent. of lampblack.
For red, 6 to 10 per cent. of red iron oxide.
For bright red, 6 per cent. of Pompeian red.
For brown, 6 per cent. of roasted iron oxide.
For buff or yellow, 6 to 10 per cent. of yellow ocher.
For green, 6 per cent. of ultramarine.

57. Seasoning Cement.—Much has been said about this subject and many specifications place a minimum age of 30 days after grinding upon cements to be furnished. The practice of seasoning cement is of European origin, and in the old German process it was customary to spread the cement out upon floors to a depth of a foot or so and then turn it over from time to time. The object of this was that in cement burned in shaft kilns at a lower temperature than the rotary kiln there is a portion of the lime in an uncombined state, and authorities claimed that by exposure to the air it absorbed enough moisture to convert what they termed *free lime* into *hydrate of lime.* An excess of uncombined lime leads to checking in the pat tests, and if in any considerable quantity, it may show deterioration in the work for which it is used. In modern practice of cement making, the danger of excess of so-called "free lime" should be avoided in the raw mixture, and if this precaution is observed and the cement properly burned, no fear need be apprehended as to the quality of a fresh cement. It is claimed by engineers and those that have made it a study that seasoning makes a more uniform product, which is very true if the process of seasoning be conducted properly, but merely specifying that a cement should be 30 days old before shipment does not remove the element of danger from a fresh cement, if there be such a danger. In most modern mills, cement is stored in heaps or bins, the heaps containing up to 50,000 barrels or more and the bins from 1,000 to 4,000 barrels or more. Cement stored in this way has no opportunity to absorb moisture from the atmosphere, except within a foot or two from the top. This can be conclusively shown by making a loss-on-ignition determination of that upon the surface and that a few feet below after a month's standing. The second sample will show little or no difference from that made when fresh, showing little or no absorption of moisture.

As there is no chemical or physical test that will disclose the age of a cement with any degree of accuracy and none that will give any indication whatever of the age of a cement from the bottom of a heap, the requirement that it should have a certain age is based entirely on the belief that it has improved in quality by standing and not on any positive knowledge to that effect. A Portland cement properly made should stand all possible tests as soon as it has lost the heat it receives in grinding, and if an age limit is placed on it by the consumers, the reason for so doing should be based on something more tangible than the simple belief that age improves it. Present methods of testing fail to substantiate this belief.

58. Sampling Cement.—This is a point to which, unfortunately, there is frequently too little attention paid. Carelessness here has frequently led to the condemning of a shipment of cement when, had proper care been taken, the same would have proved satisfactory. For instance, in testing 100 barrels, the sample should not be restricted to one sack or barrel, but should be taken from a number, and if only a series of tests is to be made, the whole should be thoroughly mixed in one sample and quartered, remixed, requartered, etc., until the desired size sample is obtained. In taking a sample from a barrel or sack, it should not be taken from the top or surface entirely, as this part, being exposed, may have absorbed moisture or carbon dioxide, and such a sample would not represent the greater bulk not so exposed. If the entire contents of the package are not thoroughly mixed, care should be taken to get the sample from the interior as well as from the exterior. A good method for this is to use a sampling auger, and if this is not at hand, to drive a pipe through the package at different points, this being withdrawn and emptied each time and a sample made of the contents. The sample obtained, care should be taken to put it into a clean vessel or bag, as this is often neglected. The sample should be tested shortly after taking, or, if kept for any length of time, should be stored in a clean, dry place where there is no great variation in temperature. It should not be left in the hot sun and then tested at 80° F. or 90° F., as temperature affects the setting time. It should not be kept exposed in a room with briquet tanks or in any other moist place, as humidity affects it. Proper care in sampling and in preserving the sample is just as essential as observing all precautions in manipulation in making tests. The number of samples and the nature of the tests on cement used in contract work is at the discretion of the engineer in charge. In some rare instances, every barrel is tested as to setting time, and every tenth or twentieth as to tensile strength; but, as a rule, about every tenth barrel is tested. A single sample from a large shipment may prove either to the advantage or disadvantage of either manufacturer or consumer; and, as it cannot fairly represent the entire lot, it is therefore not to be taken as a trustworthy indication of the quality of the entire lot.

LOCATION OF CEMENT WORKS

59. The cement industry, like every other business, owes its existence to a demand for its product. In former years it was believed that there were only a few localities where materials suitable for making Portland cement could be found. It was formerly supposed that a little section in the Lehigh Valley, Pennsylvania, about 5 miles in radius, contained the only material in the United States, and that the cement made from this was inferior to the imported article. At that time the location of a mill was governed by this mistaken idea. As the chemical nature of cement and the composition of various geological deposits became known, the cement industry began to develop and spread. Before the Lehigh Valley Portland cements were made, it was held that chalk and clay, or marl and clay, were the only suitable materials. The demonstration of the adaptability of an argillaceous limestone to this purpose caused the European process to be lost to sight for a time, and the new industry in America centered about this locality (Lehigh Valley). The increasing demand and high price encouraged competition, and the marl and clay idea was taken up in this country. As it had been successful in Europe, no other results could be expected here, and manufacturers realized that another factor became potent.

60. Proximity to the market is second only to having proper materials. Cement being both bulky and heavy, the cost of transportation figures largely in the cost to the consumer. These charges being greater than those of manufacturing, if the cement was used in the Middle West or West, led to the establishment of mills there, and now the location of a cement mill becomes a commercial problem of which the following are the factors: (1) Suitability of material. (2) Cost of manufacture: (*a*) cost of raw materials; (*b*) cost of labor; (*c*) cost of fuel. (3) Proximity to market: comparison in cost of production and freight rate with those of competitors.

1. The *suitability of material* is discussed under the heading "Selection of Raw Materials."

2. The *cost of manufacture*, as given above under (*a*), (*b*), and (*c*), would include: (1) Cost of quarrying, which would depend on the method of operation, whether by open quarry or otherwise; the cost of timbering or stripping, and cost of labor, etc. (2) Cost of transportation of the raw material to the mill. (3) Cost of grinding the raw material. (4) Cost of burning, depending on whether the material is easily or difficultly burned. (5) Cost of grinding the burned product. (6) Cost of fuel: (*a*) for power; (*b*) for burning.

3. *Proximity to Market.*—Under this head would be included shipping facilities, which would in a measure control the price of the product delivered: (1) On line of one railroad; (2) advantages of having two railroads; (3) advantages of water transportation.

In addition to the above features the cost of labor in general in the proposed locality, the question of obtaining water for boiler purposes, manufacturing supplies, etc., and all the other considerations met in manufacturing enterprises must be taken into account.

61. Portland cement is made in greater or less amounts in nearly all the countries of Europe. In the United States, the following States are represented: Alabama, Arkansas, California, Colorado, Illinois, Indiana, Kansas, Michigan, New Jersey, New York, Ohio, Pennsylvania, South Dakota, Tennessee, Texas, Virginia, and West Virginia. It is also manufactured in Mexico and Ontario. In Pennsylvania and New Jersey, argillaceous limestone properly corrected with pure limestone or clay is used; in every other locality, a mixture of chalk and clay, limestone and clay, or marl and clay is used.

SELECTION OF RAW MATERIALS

62. This is the first and most important question in connection with locating a cement mill. To know just what materials are chemically adapted to the purpose and of these which are economically adapted, is a problem deserving the greatest attention. In a general sense, there must be a source of calcium carbonate and one of silica and alumina—that is, clay. An ideal Portland cement rock is a combination of both, and is the material used largely in the Lehigh district.

63. Cement Rock.—A perfect cement rock would have about the following composition: 75 per cent. calcium carbonate, 14 per cent. silica, and 5 or 6 per cent. alumina. Magnesium carbonate, oxide of iron, calcium sulphate, and alkalies may vary, but should be in inconsiderable quantities. There are but few places in the region where this composition is approximated, but the majority of plants have a stone running from 65 to 75 per cent. calcium carbonate. This stone is dark blue to slate color, shows slaty cleavage, is rather soft, easily pulverized, and readily burned. In addition to the above it is economical, as it contains little or no moisture and is frequently run through mills without previously drying.

64. Limestone.—When limestone is used to enrich the foregoing rock, it is permissible to use a lower grade than when using it as a base in connection with clay. The best limestone should contain upwards of 95 per cent. calcium carbonate and not over 2 to 3 per cent. magnesium carbonate. A 90-per-cent. stone may be used and 85 per cent. can be used if the magnesia is within limits and silica and alumina are in proper proportions, which should be about 3 of silica to 1 of alumina or 2 of silica to 1 of alumina and oxide of iron combined. No rigid rule can be given as to the proportions of ingredients, as they may vary considerably, and yet this variation may be adjusted by the clay used. In general, however, it is well to state that limestones running above 90 per cent. calcium carbonate are far more uniform in composition than those running lower. As uniformity in the cement is one of its most essential features, uniformity in raw materials is most desirable. In addition to correct composition, a stone should be soft and easily pulverized. Stones high in magnesia are likely to prove refractory both in grinding and in burning.

65. Marl and Chalk.—The preceding remarks apply here except as to the percentage of calcium carbonate. The main feature again is uniformity. In addition, there is to be taken into consideration the amount of water to be driven off, which, in case of some marls, is as high as 50 per cent.

66. Clay.—The clay should be fine, unctuous, and should be one containing as small an amount of grit or sand as can be found. The proportion of the ingredients should be such as to balance up properly with the carbonate used. No rules can be laid down, but in general they contain about 50 to 65 per cent. of silica and 14 to 20 per cent. of alumina. The necessity for uniformity is much greater in the case of using two widely dissimilar materials, such as stone and clay, than it is in using two carbonate rocks, say one 70 per cent. and the other 85 per cent. calcium carbonate. The reason is obvious, a slight variation in the weights of each in the former case making a considerably greater variation in the product than in the latter.

USES OF CEMENT

67. As a knowledge of the nature of cement became more widely known and observations on the effect of time on constructions of this material demonstrated its power to resist all destructive influences, the applications of it became more numerous. This will be seen from the following list compiled by a manufacturer.

I. *Railroad Work:* Permanent culverts and tunnels; abutments, piers, and retaining walls; foundations for buildings and machinery; freight platforms; passenger-depot floors and pavements.

II. *Government and Municipal Work:* Irrigation and waterworks dams; conduits and sewers; street crossings; street pavements; sidewalks, curbs, and gutters; bicycle paths; retaining walls; reservoirs and cisterns; culverts and bridges.

III. *Mines and Mills:* Foundations of all kinds; boiler- and engine-house floors; waterproof pits; water-tight passages; permanent walls.

IV. *Private Enterprises:* Cement wares; fence and hitching posts and cornerstones; steps and mounting blocks; laundry tubs; burial cases; mangers and troughs; silos, sheep tubs; tannery vats; gas and water tanks; swimming pools; stucco work; vaults; bicycle race tracks; caps, sills, and columns; artificial building stone and bricks; art figures; tiles for roofs, floors, etc.; patent plaster; expanded-metal construction.

V. *Individual Use:* Floors for stables, cellars, etc.; fountains and pools; walks and driveways; fireproof construction; ornamental work; cisterns.

68. The preceding gives the more prominent uses to which cement is put, but there are also many others and new ones developing. Both corporations and individuals, in making improvements, always consider first if it can be made permanent and, if so, what is best material. No rules can be laid down as to how to apply cement in all cases, but, in general, it can be said to be used in three different ways: (1) As concrete; (2) as sand-and-cement mixture (cement mortar); (3) as neat.

The use of cement neat, that is, gauged with water and allowed to harden, is of very limited extent. The sand-and-cement mixture is more varied and of considerably greater extent, but the great bulk of cement is used in concrete work.

69. Concrete.—This is a mixture of crushed stone, slag or other hard material, sand, and cement in various proportions, according to use for which it is intended. Specifications for materials and method of mixing are not lacking. In general, it may be said that the stone or slag should be clean, sound, hard, and durable. The size of the particles depends on the use to which it is put. All of it may be required to pass a ¾-inch mesh screen, as when used for sidewalks. It may be required to pass a 2-inch or a 3-inch ring, and in heavy walls and foundations, pieces 6 inches or 8 inches in diameter or even larger may be used. The sand should be clean, coarse, and sharp. It may or may not be screened, but must be free from dirt and contain very little or no earth or loam. The cement may be of any kind or brand specified.

70. Mixing Concrete.—The mixing of concrete falls under two heads, *mixing by hand* or *mixing by machinery.* The latter is a practice rapidly coming into general use, as it reduces the cost of construction considerably. Like all new things or machine-made articles, it is fought for a time, and when its merit is established comes into general use. There are various styles of concrete mixers, of different types and patents, and at present much work is being done by them, especially on large contracts. The great bulk of concrete, however, is still mixed by hand, and many specifications are laid down for that. The following extract from the New York Central and Hudson River Railroad rules gives a method of mixing concrete by hand which will serve as an example.

71. Mixing Concrete by Hand.—Mix the cement and sand as follows: Spread about one-half the sand to be used in a batch of mortar evenly over the bed of the mortar box, then spread the cement evenly over the top of the sand, and, finally, spread the other half of the sand on top. The sand and cement should then be thoroughly mixed by turning and returning at least six times with a shovel. The mixture is then drawn to one end of the box and water poured in at the other end. Then draw the mixture down to the water with a hoe, small quantities at a time, and mix vigorously until there is a good stiff mortar. Enough water should be used that the mortar will work well under the trowel. Then level off the mixture and spread over it the required amount of broken stone or gravel, which should first be moistened: then thoroughly mix the whole mass by turning and returning in rows, at all times preserving the same thickness until the mortar thoroughly fills all the interstices.

The above method may be varied considerably, but it must be borne in mind that the one end to be obtained is as thorough and intimate a mixture of the ingredients as possible. It should also be noted that in mixing concrete no larger quantity should be used than can be worked up readily and put in work before hardening or setting begins. If a part of it hardens before being put into the work, it should not be retempered by the addition of water and worked over again, as the first set has caused the cement to lose considerable of its strength and the work in which it is used will be faulty.

72. Mixing Concrete by Machinery.—Machine-mixed concrete is preferable to that mixed by hand for the following reasons: (1) Every lot is uniformly mixed and accorded the same amount of turning as the one preceding, whereas, in mixing by hand, the thoroughness with which it is done is dependent on the workmen, one set doing it more thoroughly than the other or the same set working two lots up differently. (2) It is more quickly done. (3) It is claimed that it saves one-third of the cost of mixing. (4) Less cement is required for a given aggregate than by hand mixing.

73. Proportions of Ingredients in Concrete.—The relative proportions of the ingredients differ according to the kind of work for which it is intended and the ideas of the constructor or engineer in charge. Using the highest grade of Portland cement, the best concrete that can be made to be used under any conditions is mixture No. 1, given in Table V. Mixture No. 2 makes a concrete sufficiently strong to carry the tallest building. For ordinary construction, the proportion given in mixture No. 3 makes a good concrete. Mixture No. 4 is also a good proportion.

TABLE V

CONCRETE MIXTURES

Material	Mixture			
	No. 1. Parts	No. 2. Parts	No. 3. Parts	No. 4. Parts
Broken stone...	4	6	8	10
Sand	2	3	4	4
Portland cement	1	1	1	1

The general tendency, while it is an error on the right side, is to make a rich concrete, as many consumers, not knowing the strength of the cement, underestimate it and in doing so add needless expense to cost of construction.

Portland cement requires from 20 to 25 per cent. of water to make it work smoothly and stiffly and to insure enough for crystallization or hardening. This is for working up neat, which is seldom done; but in a 3 : 1 sand mixture, about 10 to 12 per cent. is needed. As a rule, the water is not measured, but is added a little at a time until the concrete has such a consistency that tapping with the back of a shovel brings water to the surface.

74. Impermeable Concrete.—Concrete made with Portland cement is capable, after several months, of resisting the action of the atmosphere to quite an extent, but if used at once it becomes disintegrated and is unsuitable for delicate patterns. To overcome this, several washes have been invented, which, when applied to the concrete, render it impermeable to water and atmospheric moisture. Their action is to form a coating of waterproof material over the surface of the concrete. One of the most valuable compounds for this purpose is the following:

Silicic acid	23.21%
Soda	8.90%
Potash	2.52%
Water	65.37%
	100.00%

This compound is diluted and painted over the stone or concrete and forms a thin coating of glass. It has met with excellent success.

Another method of treament is to first wash the stone with a hot solution of Castile soap, and after rubbing in and drying, to apply a solution of alum. By this treatment, the pores of the stone are filled with an insoluble alum soap.

TABLE VI

WEIGHT PER CUBIC FOOT OF VARIOUS MATERIALS

Material	Weight in Pounds Per Cubic Foot
Loose Portland cement	80 to 90
Packed Portland cement	100 to 110
Loose natural cement	50 to 60
Packed natural cement	80
Loose dry sand	About 80 to 100
Well-shaken dry sand	About 115
Loose gravel	About 100 to 120
Packed gravel	About 140
Crushed limestone	About 90 to 100
Concrete	About 140
Quicklime (dry)	64
Lime (air slaked)	Less than 64
Quartz	165
Limestone	160
Slag	110
Bricks	118 to 130
Brickwork	110 to 140

TABLE VII

NUMBER OF CUBIC FEET OF VARIOUS MATERIALS TO A TON

Material	Cubic Feet Per Ton
Clay	17
Sand	25
Gravel	27

PACKING AND SHIPMENT OF CEMENT

75. Cement is stored at the mills in bins having a capacity of from 1,000 barrels upwards, and is packed when shipped. It is shipped in cloth sacks, paper bags, or wood. Portland cement is sold on a basis of 380 pounds net to the barrel. The manufacturer's price is based on cement in bulk at the mill. If shipment is desired in barrels, the additional cost is usually 25 cents per barrel; if in cloth, 10 cents per bag (4 bags to the barrel) is added and this amount refunded if the bags are returned in good order; if in paper bags, 10 cents per barrel additional is charged and the bags are not returned. A carload of Portland cement is usually considered as 100 barrels, although 75 to 150 barrels may be shipped as a car load. Natural cement is usually sold on a basis of 300 pounds net per barrel in the East and 265 pounds net in the West. Bags or sacks contain ¼ of a barrel. Slag cement is sold at 330 pounds net per barrel.

MANUFACTURE OF PAPER

(PART 1)

INTRODUCTORY

1. History.—It is not known just how early paper was first used, but a kind of paper was made from the fiber of the papyrus plant (from which name the word "paper" is derived) by the Egyptians as early as 2000 B. C. It is uncertain at what time papyrus was superseded by paper made from cotton, but it is known that cotton paper was made by the Chinese as early as the 2d century B. C. It was not until the 8th century A. D. that cotton paper was made known to the rest of the world, when it was made by the Arabs in 704 A. D.

In Europe the art was first introduced into Spain by the Moors in the 11th or 12th century, and was made in France as early as 1189, but did not gain any headway in England until about the middle of the 17th century, after which it developed very rapidly, and at the present time paper is made by machinery from cotton and linen rags, esparto grass, straw, wood, and other vegetable fibers. The first paper mill in America was established in 1690, near Philadelphia, Pennsylvania, and to show the rapid advancement of this industry, there are at the present time over thirteen hundred pulp and paper mills in the United States and Canada.

2. The rapid progress of the paper industry is largely due to the application of chemistry, as the progress was very slow until this science came to the assistance of the

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paper maker. The importance that this branch of science has had in the advancement of the paper industry cannot be overrated.

It was the discovery of the chemist that led to the isolation of the large group of celluloses, which enter into different grades of paper. The manufacture of soda, the discovery of the bleaching action of chlorine, and the subsequent manufacture of bleaching powder, together with the discovery and application of the coal-tar dyes—were great steps in the advancement of this industry. Many other cases might be cited where chemistry has played an important part. At the present time it is necessary for a good paper maker to have some knowledge of chemistry, in order that he may understand the changes going on and be able to regulate conditions so as to obtain the best results. It is the aim to give a practical idea of the paper industry, at the same time showing the chemical changes which take place and to set forth methods for testing and keeping a check on ingredients used.

MATERIALS USED IN THE MANUFACTURE OF PAPER

3. Paper is made from the fiber of many varieties of plants and from materials that were themselves made from vegetable fibers. The examination of wood, straw, or other vegetable structures reveals the fact that it is composed of innumerable small fibers cemented together by some resinous or gummy non-cellular substance. The fibers have a cellular structure and are composed mainly of a chemical compound called **cellulose** $C_6H_{10}O_5$. This cellulose is the vegetable fiber known to paper makers as the available paper-making material, and constitutes the essential basis of all manufactured paper. The cellulose is obtained by depriving the vegetable fiber of all incrusting and cementing, resinous, and gummy matters. This is accomplished by several treatments, all of which depend on the conversion of the non-cellular constituents into soluble derivatives, the soda and the sulphite processes being the most noted.

4. Chemical Properties of Cellulose.—Cellulose that has been treated with a solution of bleaching powder at a very high temperature or exposed to this action at ordinary temperature for a long period becomes oxidized, forming oxycellulose $C_6H_{10}O_6$. This is what is known as chlorination of the fiber, which results in a loss of same, and no antichlor will restore it to its original condition. This action is very marked in treatment of straw and esparto.

5. *Schweitzer's reagent* is the only solution in which cellulose is dissolved without undergoing chemical change. This reagent is made by dissolving cupric hydrate in ammonium hydrate (.90 sp. gr.) until saturated. When cellulose is treated with this reagent, it dissolves to a thick, sirupy solution. The cupric hydrate used can be prepared by precipitation from a solution of copper sulphate with caustic-soda solution.

6. It is almost impossible to enumerate and describe in a treatise of this kind all the different materials that could be used for the manufacture of paper. Suffice it to say that almost everything in the vegetable kingdom could, under proper treatment, be reduced to a pulp. The materials taken up here will be only those brought into general use for this purpose.

7. Cotton and Linen.—These materials for some time were the only ones used in the manufacture of paper by machinery, and are still used extensively in the manufacture of the better class of writing papers. Müller gives the following analyses of these two materials:

COTTON		LINEN	
Water	7.00%	Water	8.60%
Cellulose	91.35%	Cellulose	81.99%
Fat	.40%	Fat and wax	2.37%
Aqueous extract	.50%	Aqueous extract	3.62%
Ash	.12%	Pectous substance	2.72%
Cuticular substance	.63%	Ash	.70%
Total	100.00%	Total	100.00%

Large quantities of cotton and linen rags are imported into this country, but all have to undergo a thorough system of disinfection when coming from a country where contagious diseases exist, or are likely to exist.

8. Straw and Esparto Grass.—These materials are used to a considerable extent in the manufacture of cheap grades of paper. Straw is not so valuable as esparto for the reason that it has to undergo a more severe treatment, which greatly reduces the yield of pulp. The following are analyses of straw and esparto, according to Müller:

STRAW (RYE)		ESPARTO (SPANISH)	
Cellulose	47.69%	Cellulose	48.25%
Fat and wax	1.93%	Fat and wax	2.07%
Aqueous extract	9.05%	Aqueous extract	10.19%
Non-cellulose	26.75%	Pectous substance	26.39%
Water	11.38%	Water	9.38%
Ash	3.20%	Ash	3.72%
Total	100.00%	Total	100.00%

9. Wood.—Owing to the large amount of wood available, the fiber from this material now holds the most important position in the manufacture of paper. There are a great variety of woods used for this purpose, the fiber of which is freed from the intercellular substances by either the soda or the sulphite process, which will be described later. For cheap grades of paper the wood is ground up and pulped without removing the intercellular substances. The chief woods used, together with yield per cord, and process by which the fiber is isolated, are given in Table I.

The following woods, in addition to those mentioned in Table I, are used to considerable extent: cypress, chestnut, birch, sweet gum, larch, locust, and linden.

10. Jute.—This material, which is cultivated in Bengal and consists of the bast fibers of Corchorus, is used to a considerable extent in the manufacture of paper. This fiber cannot be bleached white in the ordinary way, as the action of bleaching powder will chlorinate it. It is therefore used

TABLE I

Wood Used	Yield per Cord in Pounds		
	Soda Process	Sulphite Process	Mechanical Process
Poplar	1,000–1,500		1,400–2,000
Spruce		900–1,300	1,600–2,200
White pine			1,600–2,000
Aspen			1,600
Cottonwood			2,000
Basswood	900–1,000		
Hemlock		700–800	700–900
Jack pine			
Spruce and poplar	1,000	900–1,000	1,600–2,000
Spruce, poplar, and pine			1,000–1,200
Poplar and pine			2,000
Spruce and hemlock		1,100	
Spruce, bass, and hemlock		1,000	
Spruce and pine			1,400–2,000
Poplar, bass, and pine	900–1,000		
Poplar, bass, pine, and maple	700–1,200		
Hemlock, pine, beech, and bass	1,000		
Spruce, buckeye, and maple			1,800
Cottonwood and bass			1,700
Poplar, bass, pine, and spruce	1,000		
Bass and poplar			1,100–1,400
Aspen, poplar, and cottonwood			1,000
Poplar, pine, spruce, balsam, and tamarack			1,700
Aspen, pine, poplar, spruce, and bass			1,400
Tamarack and fir			1,700
Bass and maple	900–1,200		

mostly in the manufacture of strong wrapping paper, etc., where strength is more important than appearance. The composition of jute is shown by the following analysis:

Water	10.92%
Aqueous extract	1.43%
Fat and wax	.41%
Cellulose	62.96%
Non-cellulose	23.53%
Ash	.75%
Total	100.00%

11. Other Fibers.—Flax, hemp waste, manila, waste paper, bamboo, and the inner bark of the paper mulberry are also used to some extent in the manufacture of paper. Woolen rags are used to a moderate extent, but as it is almost impossible to bleach them, they are only mixed with other materials in the manufacture of coarse papers and wrappers.

THE MANUFACTURE OF PULP

12. General Remarks.—Before wood, rags, or other material can be made into paper they must undergo some treatment by which the fibers are separated from one another, reduced to a certain degree of fineness, and, for the better qualities of paper, all resinous and other foreign matter removed. This is accomplished in various ways, the product being known as pulp. The methods of treatment of different substances in the manufacture of pulp will here be discussed separately.

RAG PULP

13. Sorting.—Rags as they are received are unfit for immediate use. They must first undergo a preliminary treatment of sorting, cutting, and dusting before they are ready to be made into pulp.

The rags are taken from the bales and sorted as to condition of wear, color, the nature of the fiber, i. e., linen, cotton, etc., the better qualities being used in the preparation of pulp for the better class of paper. At this point of the process, buttons and other materials mixed with the rags are removed.

14. Cutting and Boiling Rags.—After the rags are sorted they are cut into pieces about 4 inches square by means of machinery. The *guillotine rag cutter*, which is adopted by quite a number of mills, will cut about 1 ton of rags per hour. The rags then pass from the cutter to the dusting machine, which is usually some form of slanting, revolving cylinder, the interior skeleton of which is provided with arms, or spikes, which revolve in the opposite direction. The cylinder being perforated, the dust passes through it freely, while the rags are removed at the other end. The rags pass from the duster to the boiler, which is either cylindrical or spherical, and is either rotary or stationary. A solution of caustic soda is used in the boiler, which insures the removal of all incrusting substances and renders the rags much more susceptible to the subsequent bleaching action. It also has the effect of softening the fibers and rendering them more flexible. The amount of caustic soda used varies, with rags treated, from 5 to 10 pounds per 100 pounds of rags. Some paper makers use lime instead of caustic soda, but this requires a larger consumption of lime than is absolutely necessary in reducing the stock, and at the same time introduces more dirt than does caustic soda, which renders it less desirable for the better class of papers. For fine grades of cotton and linen rags, stationary boilers are generally used, while for dirty, coarse stock, it is customary to use rotary boilers.

15. Bertam Boiler.—A convenient form of boiler for this purpose is the Bertam. This is a spherical boiler, 8 to 9 feet in diameter, which is made to revolve. Steam enters through the hollow journals in one side of the boiler, the steam line being provided with a cock which is kept closed

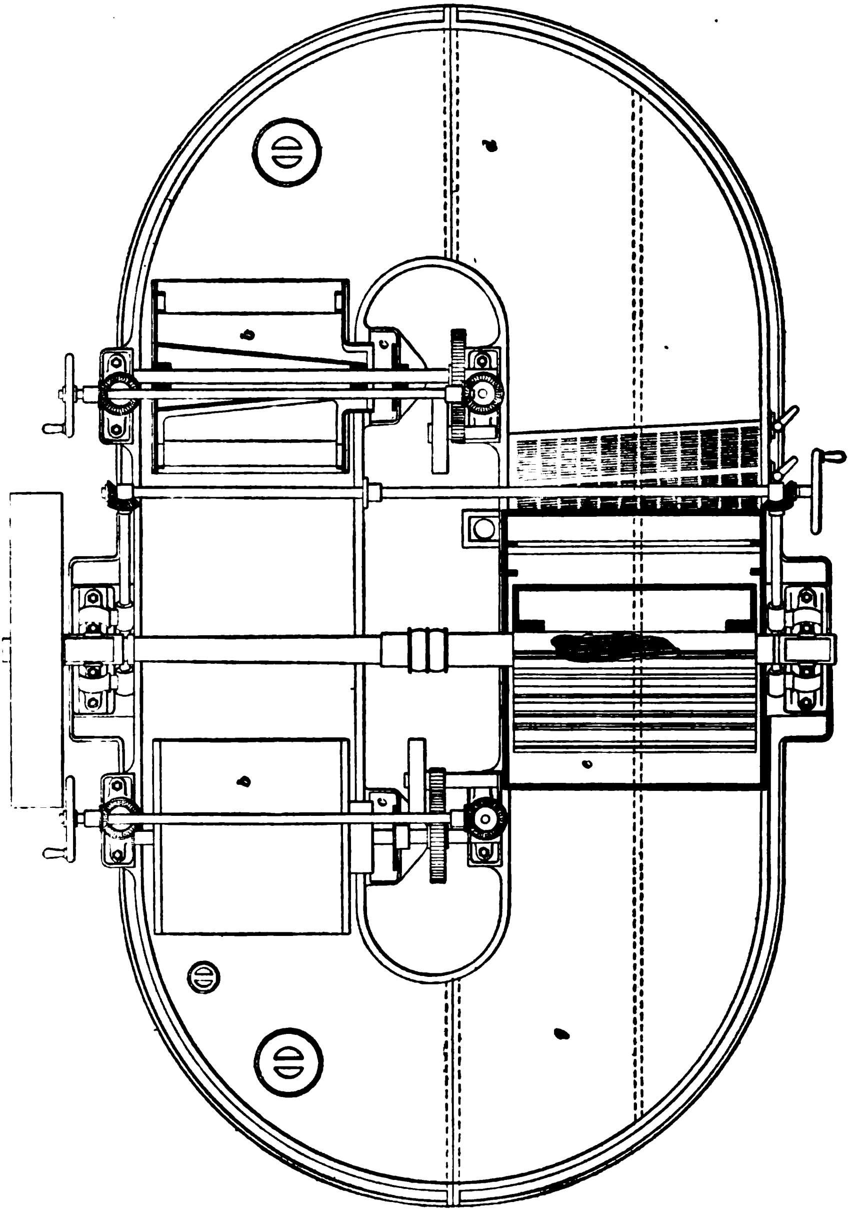


FIG. 1

until the boiler is charged. It is provided with a door on each side, through which the rags are charged, and discharged when the boiling is complete. The liquor line, which enters through the other journal, is provided with a cock also.

When the boiler is charged with rags the caustic liquor is run in, the cock closed, and steam turned on. The boiling is carried on under a pressure of from 35 to 45 pounds per square inch, depending on the treatment desired, and the time of boiling varies from 2 to 6 hours. After the boiling is complete the pressure is blown off and the spent liquor discharged by means of a cock at the bottom of the boiler. The rags are usually given a preliminary washing in the boiler.

16. Washing and Breaking.—The boiled rags are then taken to the rag engine, where they undergo the process called *washing and breaking*. In this engine the rags undergo a thorough washing, and the breaking up of the fibrous matter is accomplished.

17. Rag Engine.—The ordinary rag engine is shown in Figs. 1 and 2. It consists of an oval tank divided in the center longitudinally by a partition called the *midfeather*. In one side is situated a roll *e* bearing knives around its circumference. Immediately under this roll, in the bedplate, knives are set with their cutting edges in the opposite direction. The distance between the knives on the roll and those in the bedplate can be varied at will, regulating the degree of fineness to which the rags are cut. The floor *a* of this side of the tank is inclined and has a raised portion, shown at *f*, which keeps the rags well under the roll. On the opposite side of the tank are placed the drum washers *b*. These are cylindrical or octagonal drums having wooden ends, the circumference being covered with wire cloth, through which the water flows and passes out at *c*, the pulp being held back by the wire cloth. When washing, the tank is partly filled with water and the boiled rags are fed in at *d*. The roll *e*, revolving in the direction shown by

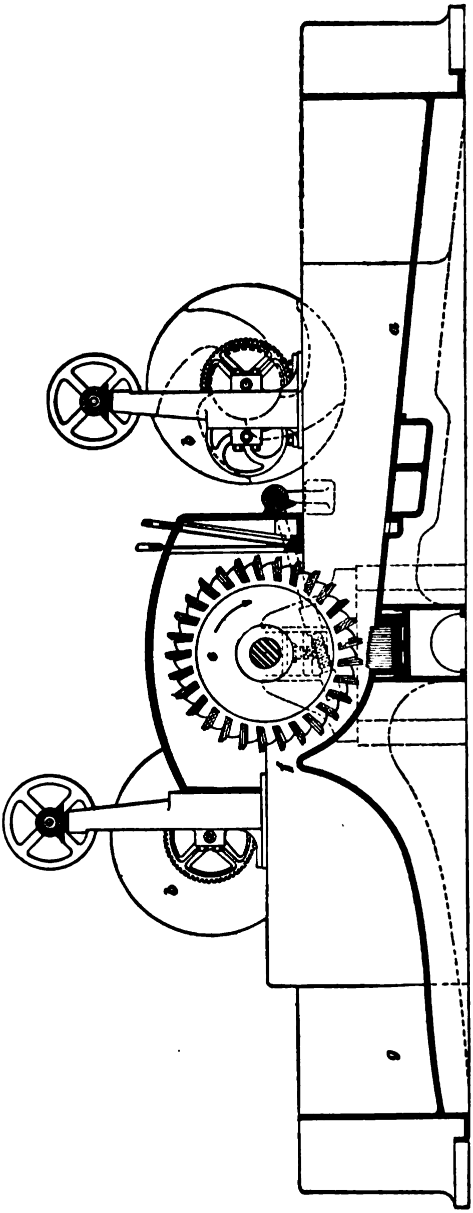


FIG. 2

the arrow, carries the rags between the knives of the roll and the bedplate, where they are cut, and over *f* into *g*, whence they pass on to the drum washers and the dirty water passes out. The supply of fresh water is such that a constant level is maintained. The washing is continued until the wash water passes out clear. The supply of water is then shut off and the washer kept in motion, being lowered as the level falls, until completely drained. The **bleaching** is sometimes done in the washer itself, but generally in what are called **potchers.** The bleaching of this fiber will be taken up later.

ESPARTO PULP

18. Esparto grass is received in bales and undergoes a treatment somewhat similar to rags. The bales are undone and the small bundles put into the hopper of a machine called a **willow,** which usually consists of several revolving cylinders with projecting teeth, which violently agitate the grass, and thereby remove the dirt, which falls below the machine, where it is removed by means of a fan. The esparto is then taken to the boilers, which are usually upright, holding about 3 or 4 tons of grass each. It is boiled in caustic soda, the strength of which is about 9½° Baumé, under a pressure of about 20 pounds. The time required is about 3 hours at full pressure. The pressure is then blown off, and, after running off the spent liquor, the grass is again boiled with water for about ½ hour, after which it is well drained and removed by means of a door at the bottom of the boiler to the washing engine. The amount of caustic soda used, the pressure at which it is boiled, and the time required all vary with the character of the boiler used and the sort of grass treated. The range is from 8 to 12 pounds of 70-per-cent. caustic soda per 100 pounds of grass; from 20 to 45 pounds pressure; and 3 to 4 hours boiling. In this boiling action, the fatty and resinous bodies are converted into soluble soaps, the silica of the grass is partially dissolved as silicate of soda, and the complex cellulose split up into cellulose on one hand and soluble derivatives on the other.

19. Washing the Pulp.—The washing is carried on in engines, similar to the rag engine, after which the drum is allowed to run until there is room for the bleach liquor, when it is added, and bleaching conducted in this engine, as will be described later.

As quite a large proportion of the fiber is lost by washing in the above washer, many paper makers object to this machine. Very good results have been obtained by using a series of tanks, arranged one above the other, fresh water entering the bottom of the upper tank, flowing from the top, and entering the bottom of the next lower tank, and so on, the motion of the water being very slow, so that the finer particles are not carried away.

STRAW PULP

20. Straw requires a more severe treatment than esparto or rags, as the knots in the straw must be reduced, which necessitates the use of an excessive amount of soda. By this treatment the finer fibers are liable to be carried away with the wash water, and yield has been as much as 10 per cent. less than that of esparto. Wheat, oat, rye, and barley straws are used generally, wheat and oat forming a large bulk of the material used in this country. From 10 to 20 pounds of caustic soda per 100 pounds of straw are required to thoroughly boil it.

The straw is first cut into short lengths by means of a cutter, similar to the rag cutters, conveyed through a wire cylinder, where the dust is removed, and thence to the boiler, which is usually of a cylindrical, rotary form; but some paper makers prefer to use the stationary boilers. The boiler is first partly filled with the caustic liquor, on account of the straw being so bulky. The straw is then added until the full amount has been put in, the balance of the liquor run on, and the head of the boiler screwed in its place. It is cooked for from 3½ to 8 hours at a pressure of from 20 to 40 pounds. The strength of the liquor varies from 6° to 10½° Baumé. When the boiling is complete, the boiler is allowed to cool and the charge to run through a pipe to the drainer tanks. These are large tanks, provided with perforated bottoms, in which the liquor is drained out and the stock washed, to some extent. It is then transferred to the potcher, where it is again washed and bleached.

21. A low grade of pulp, suitable for the manufacture of the coarser grades of paper, such as wrapping paper, is made from such materials as *jute*, *manila*, *flax*, and *hemp waste*.

The materials are prepared by cutting and boiling by processes very similar to the treatment of esparto and straw, with slight modifications. Jute is boiled with a solution of lime instead of caustic soda.

The treatment of the above materials is not so particular as is necessary in the manufacture of better grades of pulp.

WOOD PULP

22. By far the greater quantity of paper used at the present day is made from wood pulp, wood being an abundant and cheap raw material. There are three processes in common use for the manufacture of wood pulp, the *mechanical*, the *soda*, and the *sulphite*. The two latter are chemical processes in which the intercellular substances are dissolved and removed by the aid of certain solvents. In the mechanical process the intercellular substances are not removed and as a consequence the paper made from this pulp is of a poorer grade.

THE MECHANICAL PROCESS

23. Grinding the Wood.—Wood is made into pulp in some mills by **mechanical** means, in which case none of the intercellular substances are removed. It is, therefore, a very difficult matter to bleach such pulp, and it is mostly used in cheap papers, such as for newspapers, etc. There are various methods of preparing this pulp, but they all consist in pressing the wood against a rough stone, upon which water is kept running. Fig. 3 shows a wood-pulp grinder from which good results are obtained. The sandstone, which is grooved and pitted to make it as rough as possible, is enclosed in the iron case *a*. The wood (the knots being cut out) is cut in short lengths and put in at the openings *b*, after which the doors are closed. The wood is kept tightly pressed against the revolving stone by means of the hydraulic presses, during which operation water is continually running on the stone. This stream of water carries the pulp along a runway, where it passes through a rake, which holds back large pieces of wood, and thence to the sorters, which consist of cylinders covered with wire cloth. The coarser fibers that are retained by the cloth are further ground up by the refiners, which are a couple of sandstone cylinders, the upper one of which revolves. After leaving the refiners, it is passed through the screen again.

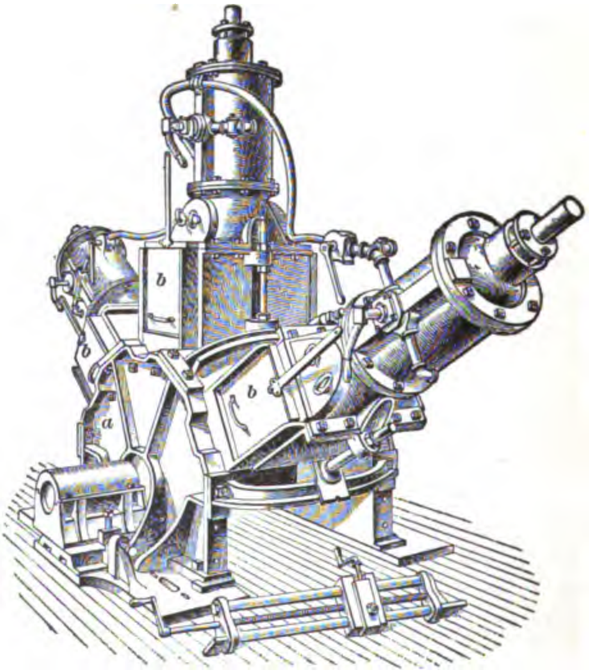


FIG. 8

Wood is sometimes *steamed* before going to the stone, which results in the production of longer fibers. The pulp that has been ground and sorted is run into a vat, and thence taken up by a cylinder covered with wire cloth, revolving in the vat, and transferred to an endless felt, which in turn carries it to a large roll, around which it is wound until the required thickness is obtained, and then cut from the roll and folded. There is much room for improvement in the methods now in use for the preparation of ground wood.

The wood most generally employed is pine. Aspen and cottonwood are also used to a considerable extent.

THE SODA PROCESS

24. By the soda process the acid compounds making up the intercellular substances of the wood are brought into solution as salts of soda, which result is accomplished by treatment of the wood with caustic soda. On account of the strong solvent power of the caustic liquor, it is not necessary to remove the knots or rotten portions of the wood.

25. Chipping the Wood.—The bark is peeled from the wood, which is then cut into pieces about 5 feet long and .75 feet thick. These pieces are conducted to the chipper on small cars. The chipper, Figs. 4 and 5, consists of a large revolving cast-iron disk, which has three knives *b* bolted to

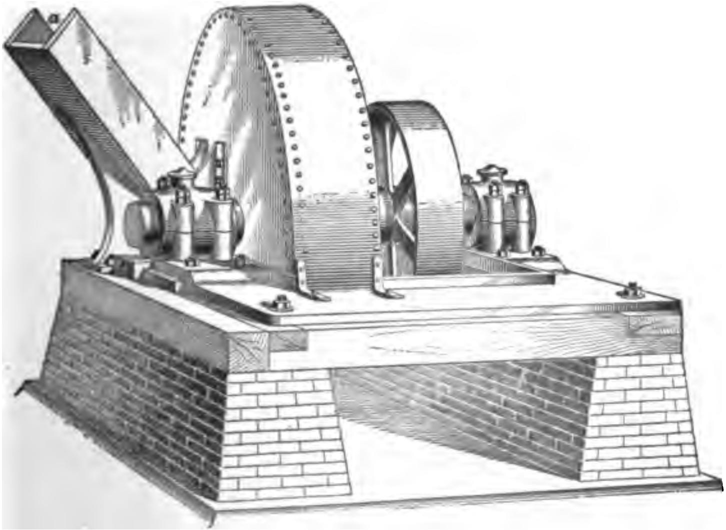


FIG. 4

it in a slanting position at equal intervals. The knives are so set that the blades project; the size of the chip, which ranges from ¼ to ¾ inch, is regulated by the setting of the knives.

The large sticks of wood are taken from the cars and introduced through the trough *a*, which is set in a slanting position, so that the stick will move forwards by means of its own weight. The disk revolves at a high speed, and the sticks are cut into chips across the grain. The chips are caught and conveyed to the digester room by means of an endless chain of buckets, where they pass through a mechanical screen, which removes the dirt and fine particles, and thence to the digesters. It requires from 10 to 15 minutes to chip 1 cord of wood.

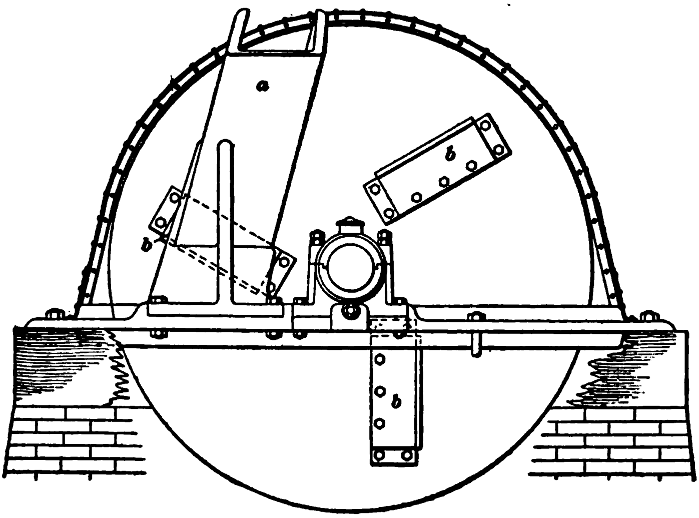


FIG. 5

26. Digesters.—These are either rotary or stationary, and cylindrical or spherical. It seems to be a question as to which form is the most common, it being almost equally divided between the upright cylindrical stationary and the horizontal cylindrical rotary. The rotary form is heated by

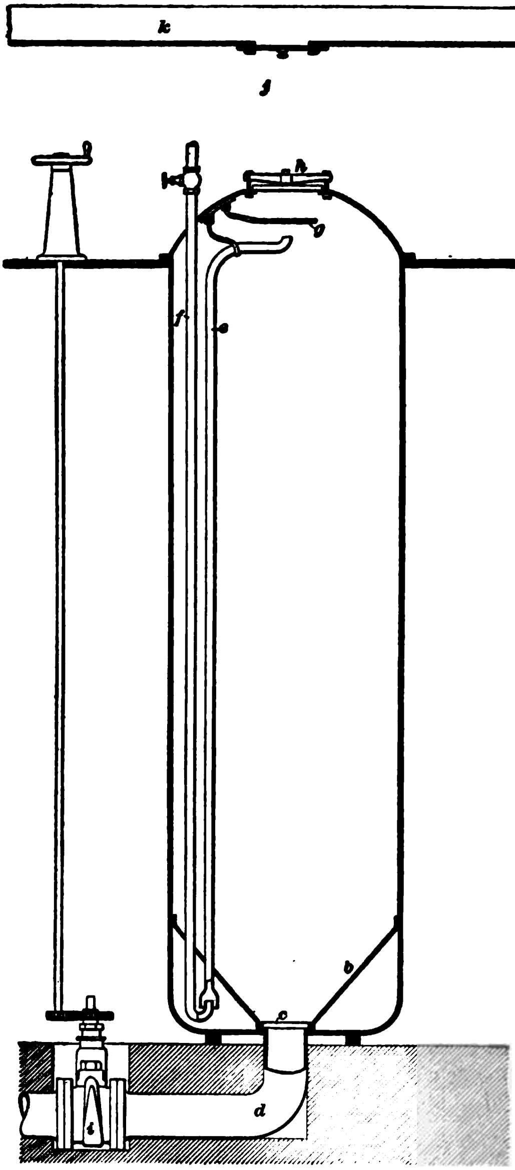

k
j
h
g
f
e
b
c
d
i

FIG. 6

coils, supplied with steam through the trunnions. The upright digesters are heated by live steam, by direct fire, or by steam jacket, the greater number being heated by live steam.

Good results are obtained by the use of an upright cylindrical digester, 27 feet high by 7 feet in diameter, the top of which is semi-spherical. There is an opening at the top, through which the chips and liquor are charged, after which a plate is bolted down tightly over the opening before turning on the steam. This digester is heated by live steam, the steam line entering the top and extending to the bottom, where it is discharged through a steam ejector so constructed as to keep the liquor in constant circulation through the arrangement for that purpose. There is a perforated plate at the bottom of the digester, which surrounds the outlet but does not cover it. The liquor passing through this plate is conveyed by the circulating pipe to the top of the digester and discharged again over the wood. This circulation is kept up throughout the entire cooking operation.

27. Fig. 6 shows a cross-section of an upright cylindrical digester, in which *b* is the perforated plate extending from the side of the digester to the ring *c*, surrounding the outlet into the dump line *d*. The plate allows the liquor to pass through, while the stock is held back, after which the liquor is conveyed by means of the circulating line *e* and steam line *f* to the top of the digester, where it is discharged against a plate *g*, which sprays it over the wood again. The plate *g* and the top of the circulating line *e* are held by brackets, and can be removed to one side while filling the digester. The digester is charged through the manhole *h* by opening a slide *j* in the conveyer box *k*, which extends along over all the digesters and carries the chips on an endless chain. The pipe through which the liquor is run into the digester can be swung to one side when the required amount of liquor has been added.

After the digester is charged with chips and liquor the plate is bolted down securely over the manhole and the steam turned on. A continuous circulation is kept up by the steam discharging up the circulating line and thus drawing up the liquor that has collected. The gate valve *i* in the discharge, or dump line, can be opened or closed from the top floor of the digester room.

28. Digesting or Cooking.—One of these digesters holds about 4¼ cords of wood. The liquor used is caustic soda, the strength of which varies from 10° to 15° Baumé at 60° F. and a causticity from 92 to 94 per cent. It requires from 4,000 to 5,000 gallons of this liquor, depending on the kind of wood used, the condition of same, and the strength of the liquor used. The time required for each cook is from 6 to 10 hours at full pressure, depending on above-mentioned conditions. It is customary to cook at a pressure of from 100 to 120 pounds, taking from 2½ to 3 hours to get up pressure. When the cooking is complete, the pressure is blown down to about 75 pounds, after which the gate valve is opened at the bottom of the digester and the pulp forced up through the discharge line into the blow tank, where it strikes with great force against a dash plate, which separates the fibers. (This operation is termed the *blowing of a digester.*) When blowing down pressure, it is discharged in this tank also. It is generally situated over the building in which the stock is washed. The yield per digester depends on the wood used, as given in Table I. The pulp after cooking is of a light-brown color, while the liquor is dark brown, verging on black. This liquor, which contains about all the alkali combined with the acid products of the wood, is termed *spent liquor.*

29. Washing the Pulp.—The next step in the process is the washing of the stock, usually accomplished in large wash pans, which hold one digester of stock. These pans have a perforated false bottom, through which the liquor and wash water pass freely, while the stock is held back. The stock to be washed is dumped into the pan from the blow tank, leveled with a long rake, and the liquor allowed to drain off, after which it is washed for a short time with weak, spent liquor, and finally with hot water. The liquor and washings are caught in tanks below and used in the recovery of soda, which will be discussed later. When the wash water is reasonably clear, the stock can be considered washed, and is ready to pass through the screens and over the wet machines, or through the washers to the bleachers. The object in washing first with weak liquor is to wash the stock with as little water as possible, as all the water added to the liquor must be again removed in the recovery process.

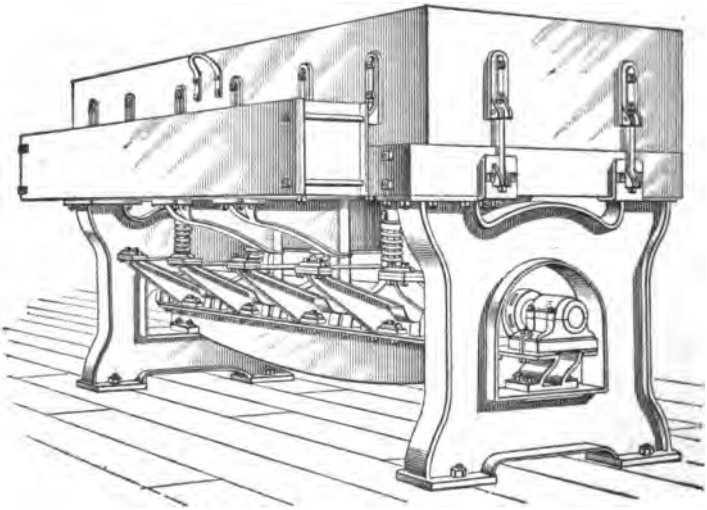


FIG. 7

The liquor and washings are run into what is termed the *strong tank* until they become too weak to be used economically in the recovery department, after which they are run into the weak-liquor tank to be pumped up and used for preliminary washings of other pans. After the stock has been completely washed, the plug at the bottom of the pan is removed, which is accomplished by means of a screw arrangement at the top of the pan. The stock is then washed down into the dump line by means of a rubber hose, which throws a strong stream of water. From the dump line it is pumped up into the screens, where all material which has not been acted on in the process of cooking is removed.

30. Screens.—The screens used are of various makes, but have about the same form and accomplish the same purpose. In Figs. 7, 8, 9, and 10, which show the "Success" screen, the general idea is plainly set forth. Fig. 7 gives a general view of the screen, together with the flow box. Fig. 8 shows the screen box open, exposing the wooden diaphragm top plates *a*, the rubber diaphragm *b*, and the apertures *c*, through which the stock passes in going to the flow box *d*. Fig. 9 shows the diaphragm top plate *a*, the rubber diaphragm *b*, the wooden diaphragm *c*, the iron diaphragm *d*, the wooden pitman *e*, the spiral spring *f*, the spring plank *g*, the cam oil box *h*, the wooden shoe *i*, the pitman head *j*, the shoe clamp *k*, the bridge tree *l*, the cam *m*, the journal *n*, the ring oiling pillow-block *o*, and the screen shaft *p*.

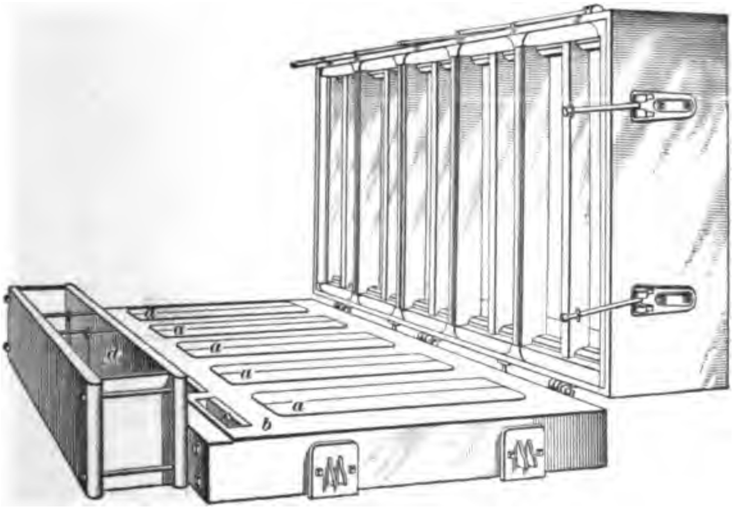


FIG. 8

Fig. 10 shows one of the brass screen plates, the slits in which are very fine, to admit of the passage of only well-cooked fiber, holding back material that has not been acted on, etc. These screen plates should be watched very carefully, and when there are any openings large enough to allow this slivery substance to pass through, the plate should be changed.

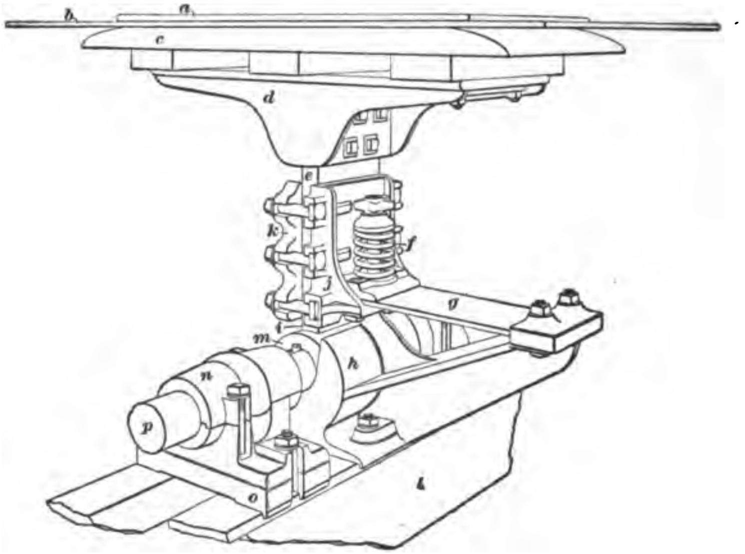


FIG. 9

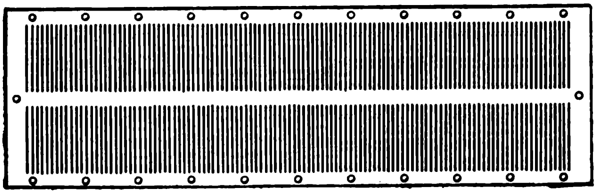


FIG. 10

In the screening operation, the pulp is pumped on the screens in a thin, watery suspension, when it is carried through the screens by the following action: The screen shaft *p* is absolutely round, but the cam is made square with the corners rounded off, so that in every revolution of the shaft there are four upward and four downward movements of the shoe, which movement is communicated to the rubber diaphragms, which gives the screen its suction. When the shaft is running at full speed it makes from 165 to 175 revolutions per minute, which will cause from 660 to 700 vibrations of the diaphragm per minute. At each downward stroke the pulp is sucked through the screen plate, there being a space of 4 inches between the screen plate and the top diaphragms, and thence it runs through the apertures at the side of the diaphragms and out through the flow box. The flow box is provided with a partition, which causes the out-flowing stock to stand at such a level that the apertures in the diaphragms are always covered, otherwise the screen would lose its suction, by air coming back through the flow box, and refuse to act. In case the screen refuses to act and flows over, the stock is shut off and water turned into the flow box, after raising the partition, until it rises to the screen plates in the screen, which will drive out all the air, and the screening can then be continued as before. Good results are obtained by the use of the screen mentioned above.

31. The **Gotham screen** is built somewhat similar to the above screen. The advantage claimed for this screen over other makes lies mainly in the simplicity of its construction. The rod that carries the wooden shoe running upon the surface of the cam, on the cam-shaft, and attached to the iron diaphragm on the other end, comes as near to being as solid a casting as possible, having a taper-fit joint at both ends. This portion of the screen, when the shaft is running up to its proper speed, 175 revolutions, is vibrating at the rate of 700 times per minute. A great advantage is derived by having this shaft practically in one piece, as it eliminates the use of bolts and nuts, which have a tendency to work loose on account of this rapid vibration. The cam on this screen is so adjusted that the upward stroke is quick and of shorter duration than the downward stroke; and if slivers, etc. are sucked into the slits of the screen, the quick upward stroke will almost invariably clean the plates of such obstructions. The stock is taken away through apertures arranged along the side of the diaphragms. These apertures discharge into spouts, which run crosswise of the screens underneath the frame and connect with the flow box. By this arrangement there is ample room for the stock to get away from the diaphragms; and on account of the fact that it is taken down to the level of the spouts underneath the frame, the suction on the screens is much assisted.

The Gotham screen is very much used by paper makers and produces good results. The excessive amount of water is removed from the pulp before going to the bleachers by either passing over a wet machine or through a long trough in which a number of washers are revolving.

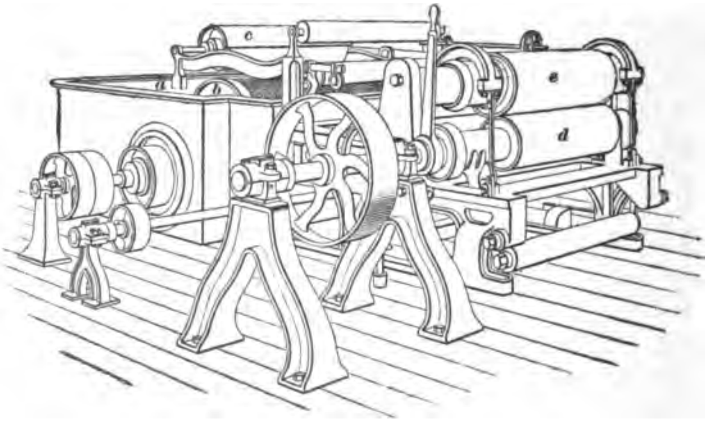


FIG. 11

32. Wet Machine.—When using, as shown in Fig. 11, the stock from the flow box passes into the vat *a*, in which a roll *b* covered with wire cloth is revolving. This roll takes up the stock and transfers it to an endless felt, which runs over the couch roll *c*. The stock is carried by the felt between the rolls *e* and *d*, the felt passing around the roll *d* and back to the couch roll, while the stock clings to roll *e*. In some mills it is allowed to collect until it reaches the required thickness, when it is cut from the roll, folded, and transferred to the bleachers or shipped as unbleached fiber. However, it is generally removed from the roll as fast as it is formed, by means of a mechanical arrangement, which rubs against the full length of the roll *e*, cutting the pulp off and allowing it to drop upon a slanting board. This board guides the stock to an alley belt, which conveys it to the bleachers.

33. In case the water is removed by means of washers, the process is as follows: The pulp is run directly from the flow box to a long washer box, which has a number of octagonal drum washers, situated at short intervals along the whole length of it, and are all rotated by means of an endless chain. These washers, Fig. 25, take up the water, which passes out at the axis of the drum, and is conveyed away by means of a small trough, along the side of the washer box. When the pulp reaches the other end of the washer box it is very thick and can be then conveyed to the bleachers.

34. Liquor for the Soda Process.—The liquor is made up in pans, usually about 10 feet by 10 feet, which have agitators extending to the bottom, and worked by means of a friction clutch and gearing at the top. The pans are also provided with siphon pipes, which can be raised or lowered, and steam lines running to the bottom. The pan has an opening at the bottom for washing out the sludge into the sewer, which, when making liquor, is closed with a tight-fitting plug. There is a water-line, a weak-liquor line, and a line from the leachers, emptying into each pan, all of which are provided with cocks.

When making up a pan of liquor, the leacher-line cock is opened (the leaching system is explained under "Recovery of Soda"), the recovered liquor allowed to flow in, and the agitator started. The liquor maker makes a test of the liquor from time to time while it is coming up, to ascertain its density by means of a Baumé hydrometer. If he finds that the pan is coming up too strong, the weak-liquor pump is started and the cock on that line opened up. By practice, the liquor maker is able to so regulate the running of the liquor that there will not be a difference of more than ¼° Baumé in the finished pans. If, when the pan is filled up to a certain mark, the liquor does not come up to the desired strength, fresh soda is added to accomplish this end. When aiming at a certain strength of caustic liquor, the carbonate liquor should be made about 2½° Baumé stronger than the strength of caustic liquor required. The liquor is causticized to convert the sodium carbonate to the hydrate, by the addition of lime, which is generally put into an iron cage hanging down into the liquor. The lime should be added cautiously, to avoid boiling the pan over. After the required amount of lime has been added, the steam is turned on cautiously and the pan allowed to boil for about ½ hour, agitating all the while, after which the agitator is stopped and the sludge allowed to settle. The carbonate of soda is converted to caustic soda by the addition of caustic lime, according to the following formula:

$$Na_2CO_3 + Ca(OH)_2 = 2NaOH + CaCO_3$$

The lime sludge settles to the bottom, leaving the caustic liquor perfectly clear, provided the recovered ash was well burned. From 6,000 to 7,000 pounds of 58-per-cent. soda is used to make one strong pan, depending on the size of the pan and the strength of the liquor required, and in practice from 625 to 650 pounds of a good caustic lime are required to causticize 1,000 pounds of soda ash.

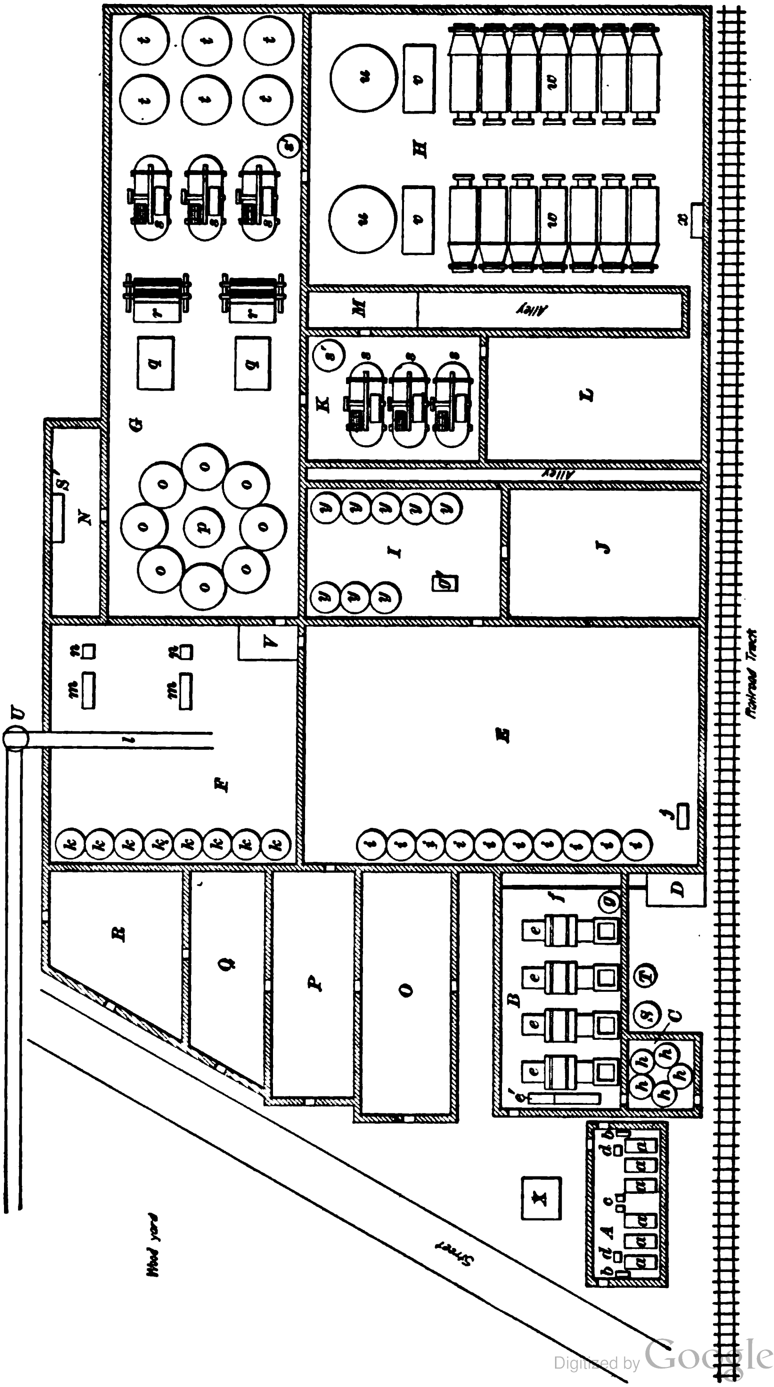
35. It is customary to have two tanks in the cellar of the alkali room, one for the strong liquor and the other for the weak. The former is used for storing strong liquor for the digester room, while the latter is used for storing second and third washes, which liquor is used in making up the first wash, and when crowded, is pumped through the leachers in place of water. In some mills each wash is kept in a separate tank and used in making up the wash preceding it in the next tank.

The washes are made as follows: When the strong pan has settled sufficiently, the siphon pipe is lowered and the liquor siphoned into the strong tank in the cellar. (It is customary to run a strong pan and the first wash of another pan at the same time, in order that they will mix in the right proportions in the strong tank in the cellar.) When the liquor is all out of the strong tank and the siphon pipe down to the sludge, the pipe is raised, the agitator started, and the pan pumped up with weak liquor, by means of a centrifugal pump in the cellar. (It might be well to note here that the centrifugal, or fan, pump is made use of in almost every department.) It is the aim of the liquor maker to make as large a first wash as will be carried by a full, strong pan, and make liquor of the required strength for the digesters. The second and third washes are made similar to the first and are run down into the weak-liquor tank, to be used as stated. The plug is then drawn, the sludge washed out into the sewer, or over to the lime reclaimer, where it is well drained and burned back to caustic lime and used again. It is customary in most mills to keep samples of the strong liquor and each of the washes made, which are tested morning and evening by the chemist in charge for Baumé strength.

36. Ground Plan of a Typical Soda Pulp Mill.—Fig. 12 gives the relative position of buildings and machinery in a typical soda pulp mill. *A* is the Yaryan room; *a*, the Yaryan evaporators; *b*, the vacuum pumps; *c*, the lead pumps; and *d*, the tail pumps.

B is the rotary room; *e*, the rotary furnaces, with fireboxes and flues; *f*, the line shaft; *g*, the fan blower; and *e'*, the ash conveyer.

C is the leacher room; *h*, the leaching battery. The black-ash tank is located over the battery, into which tank the ash is carried by the conveyer mentioned above.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V
X
Alley
Alley
Railroad Track
Street
Wood yard
a
b
c
d
e
e'
f
f'
g
g'
h
i
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l
m
n
o
p
q
r
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FIG. 12

D is the lime shed, under which is situated the rotary engine.

E is the alkali room; *i*, the causticizing pans; and *j*, the scales. The balance of the room is generally kept filled with a stock of soda ash in bags of convenient size to handle. The storage tanks are in the cellar under this room.

F is the ground floor of the digester and chipper room; *k*, the digesters; *l*, the track for hauling wood to the chippers; *m*, the chippers; and *n*, the conveyers. The chippers are on the ground floor, and the room is all opened up to the top floor of the digester room, there being a platform *V* to get from the alkali room to the wet-machine room.

G is the wet-machine room; *o*, the wash pans; *p*, the blow tank, which is above the wash pans; *q*, the screens; *r*, the wet machines; *s*, the bleachers; *t*, the drainers; and *s′*, the tanks of bleach liquor for bleachers. Strong- and weak-liquor tanks are under the wash pans.

H is the machine room; *u*, the chests of bleached and drained stock for the machines; *v*, the screens; *w*, the pulp machines; and *x*, the scales.

I is the bleach room; *y*, the bleach mixers; and *g′*, the stairs leading to the cellar, where the bleach liquor is stored.

J is the bleach storeroom.

K is a room in which there is another set of bleachers.

L is the pulp storeroom.

M is the pulp-machine engine room.

N is the chipper and conveyer engine room; *S′*, stairs leading up to top floor of the digester room.

O is the boiler room; *P*, the engine room; *Q*, the supply room; and *R*, the iron storeroom.

X is the blacksmith shop.

S is the weak-liquor tank for the Yaryans.

T is the strong-liquor tank for rotaries.

There is an alley between *I*, *J* and *K*, *L*; and also one between *K*, *L*, and *H* for the purpose of furnishing light to these rooms.

The laboratory and wood yard are situated across the road. The wood is conveyed in cars, with the assistance of the turntable *U*, to the chippers.

THE RECOVERY OF SODA

37. Evaporation of the Liquor.—The strong liquor from the wash pans is pumped over to the Yaryan evaporators, the section, plan, and elevation of which is shown in Fig. 13 (*a*), (*b*), (*c*), and (*d*), in which (*a*) is the plan, (*b*) a front elevation, (*c*) a side elevation, and (*d*) a longitudinal section of an evaporator showing circulation. The operation of evaporating the liquor is as follows:

Steam, which may be exhaust steam from the engine or live steam direct from the boilers, is led into the cylindrical chamber surrounding the coils in the first effect by the pipe *s*. The liquor to be concentrated, which should test from 8° to 10° Baumé at 60° F., is fed into the first tube *e* of the return-bend coils of the first effect in a small but continuous stream. It immediately begins to boil violently, becoming a mass of spray, containing, as it advances through the heated coils, a constantly increasing proportion of steam. The inlet end of the coil being closed to the atmosphere, and steam being continually formed, the contents is propelled through the tubes at a high velocity, finally escaping from the last tube of the coil into the separator *a*. Here the steam, or vapor of evaporation, and liquor carried with it are discharged with great force against the baffle plates *f*, which separate the liquor from the steam, causing the liquor to fall to the bottom and allowing the steam to pass off through the ingeniously contrived catch-all *b*, as shown by the direction of the arrows in (*d*), which effectually removes any liquor still remaining in the steam and conveys it into the liquor line coming from the first effect. The liquor from the first effect is led to the back of the second effect, where it enters the coils, and the same operation is performed as in the first effect, and so on through the entire system, the liquor being constantly reduced in volume. The steam from the liquor in the first effect passes into the second effect at *g*, and surrounds its coils. The steam from the final effect goes to the condenser *c* and vacuum pump *h*, a high vacuum being maintained in the separating chamber, and, consequently, in the coils. Hence, the boiling point of the liquid is at a lower point of temperature than that of the surrounding steam, and by the condensation of steam

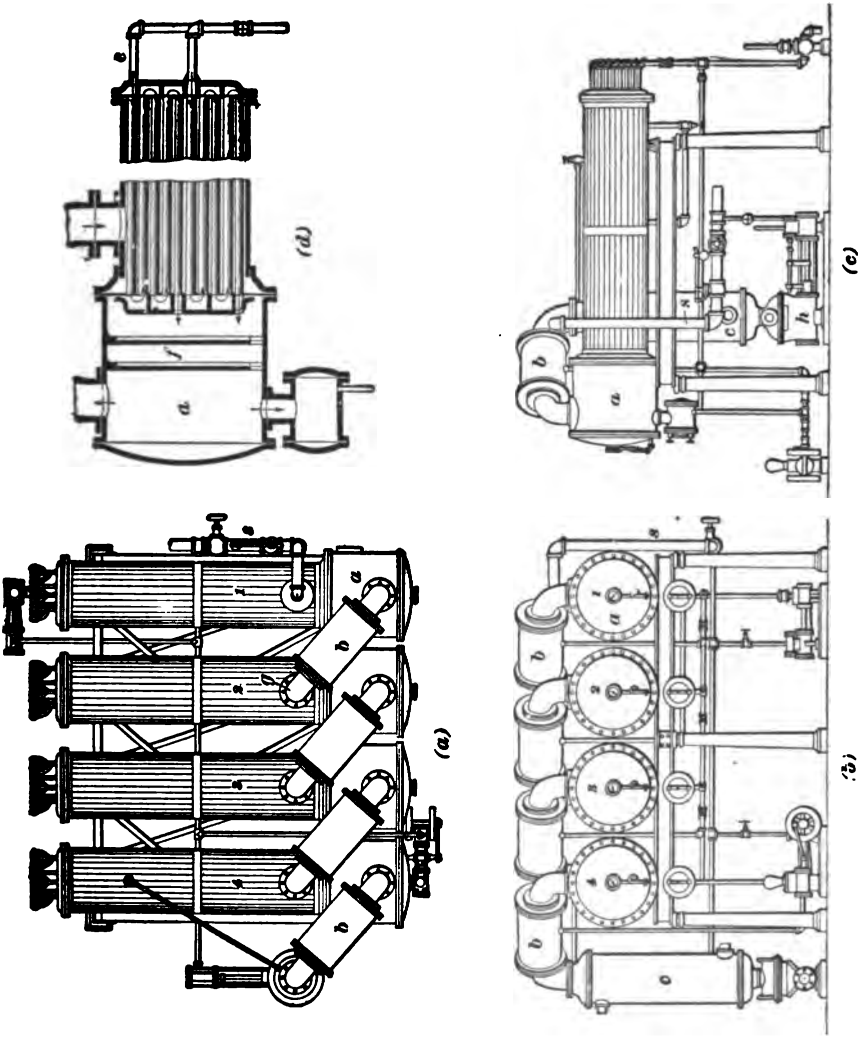
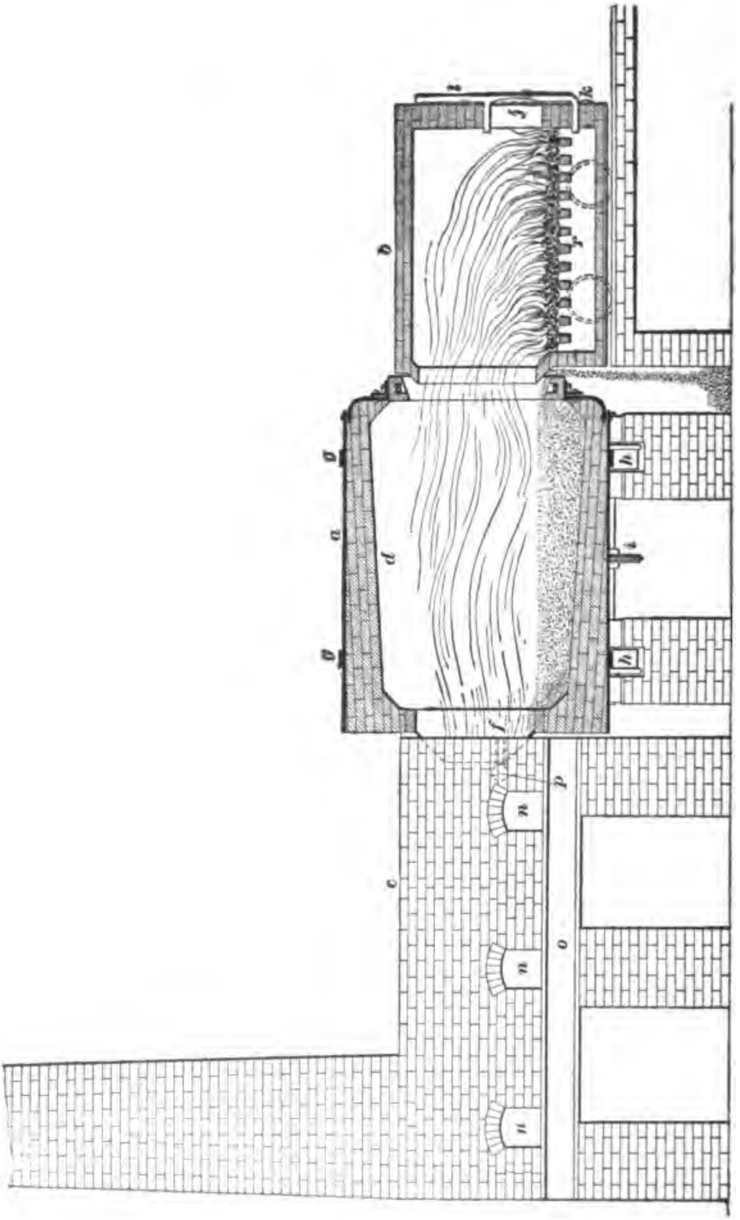


FIG. 13

from the previous effect, upon the cooler pipes in this effect, a vacuum of a less degree is maintained in the next succeeding


b
i
k
f
p'
g
a
g
d
h
i
h
f
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c
n
n
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o

FIG. 14

effect. This relative reduction in pressure, and consequently in boiling temperature, automatically adjusts itself, however many effects are used, thus accomplishing the boiling of the liquor by the steam produced by its own evaporation in the previous effect. The only steam required to be supplied is that of the first effect, which varies in different mills from 20 to 40 pounds. The liquor entering at about 8° or 10° Baumé at 60° F. leaves the Yaryan at about 38° Baumé at 60° F.

The evaporated liquor is then conducted to a tank, from which it is supplied to the rotary furnaces, where it is dried and calcined.

38. Rotary Drying Furnaces.—Fig. 14 gives a typical rotary furnace, with movable firebox; also flue, cross-section of rotary and firebox, and elevation of flue.

In the figure, *a* is the rotary; *b*, the firebox, and *c*, the flue. The rotary is lined with firebrick *d*, so as to form a conical interior, the brick being held more firmly in place by the face irons. The rotary is surrounded by tracks *g*, which run on flanged wheels *h*, the flanges being on the opposite sides of the two wheels, and thus holding the rotary firm. The wheels, and consequently the rotary, are made to rotate by means of a sprocket *i*, which connects by means of an endless chain with a sprocket on the line shaft. The liquor line passes through the wall of the flue at *p*, and thence into the back of the rotary at *f*, the rotary revolving around the line. The concentrated liquor entering through this opening in the back of the rotary passes on through, becomes thicker and thicker, and finally ignites. By the time it reaches the front of the rotary, all the resins, etc., taken from the wood have been burned.

The fuel is charged into the firebox through the door *j*, and the ashes are removed at *k*. The grate bars *r* can be removed from time to time and cleaned. The blow pipe *l* coming from the fan blower enters the furnace above the door and also under the grate bars, thus driving the fire back into the rotary. The firebox is mounted on wheels and can be moved back when repairing the rotary.

The flue is built of brick and is mounted upon iron supports *o*, which are in turn mounted upon brick pillars. This flue is so arranged that considerable ash, which is carried back into it by the draft, can be removed through doors *n* along its side. The back end of the rotary is conical and projects into the flue.

39. The method of conveying the ash from the rotaries differs in different mills; some convey it by means of an endless chain to a tank over the leachers, while others allow it to drop into a car placed below and at the end of a chain running along the front of the rotaries; when full, it is conveyed to the leachers and another car put in its place.

40. Leaching.—The black ash is leached by several different methods. In most mills the ash is spread out in large, shallow pans, with perforated false bottoms. It is then thoroughly washed with hot water, by which the carbonate of soda is leached out and conveyed to the causticizing pans. Some mills employ a leaching battery, which consists of a series of iron shells with perforated false bottoms, which surround but do not cover the outlet at the bottom, as shown in Fig. 15. The shells are so connected by means of necessary piping that the water used in leaching passes into the top of the weakest shell, and from the bottom of this shell into the top of the next stronger, and so on until it reaches the last shell filled, after which it passes into the causticizing pans. Any one of the shells can be turned into the line going to the

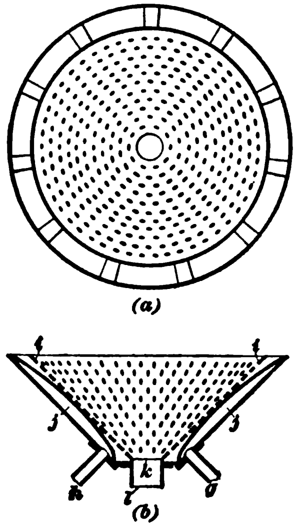


FIG. 15

causticizing pans and any one can be cut out of the battery when desired.

41. In some mills the shells mentioned are connected in circle, in which case the ash is carried by an endless chain to the tank over the center of the leaching battery, from which it can be dumped, by means of a pipe, into any one of the shells. In other mills they are piped up in a row. The ash is then taken up in cars, which are run on a track over the leachers and dumped into any one of them.

Fig. 15 gives two views of the bottom of a leacher; (*a*) is a section looking down upon it and (*b*) a vertical cross-section through the center. At *h* is shown the circulating line; *g*, the line leading to the alkali room; *i*, the perforated false bottom; *j*, the space between the false bottom and side of the shell; *k*, the ring in which the plug fits, and *l*, the dump line.

42. When filling a shell, a plug is securely placed in the bottom of same and the ash dumped in until about three-fourths full, when the cap is placed on the manhole at the top and screwed down tightly. The hot water is then pumped into the weakest shell until a pressure of from 45 to 60 pounds is reached in the battery. The cock is then opened in the line leading from the last shell filled to the causticizing pans. The leaching is then continued, as above suggested, until the pan is up, or liquor coming from the last shell tests only about 1½° Baumé, when the weakest shell is cut out, dumped, filled with fresh ash, and the leaching continued as before.

43. Calculation of Recovery.—Black ash contains from 43 to 48 per cent. Na_2O. When figuring recovery, in some mills the black ash made is weighed and recovery calculated from the amounts of each ash used in making up the liquor. The recovery is usually calculated by multiplying amount of 48-per-cent. soda necessary for one digester by the number of digesters put on, subtracting amount of soda used in the alkali room (after converting the latter to 48-per-cent. Na_2O) and dividing the remainder by the total amount used, as above calculated. It varies from 75 to 90 per cent. in the different mills.

The sources of loss of soda through the mill are (1) in the dump of lime sludge from causticizing pans; (2) blowing of the digester; (3) imperfect washing of stock in the wash pans; (4) up the chimneys of the rotaries; (5) formation of silicate of soda due to SiO_2 present in the lime; and (6) leaching the black ash, under which head will also come loss due to imperfect burning, as this is very frequently the cause of a considerable loss of soda.

The caustic liquor used in boiling straw and esparto is also recovered by a process similar to the above.

THE SULPHITE PROCESS

44. The **sulphite process,** which from its present use should be properly termed **bisulphite process,** was first worked on a practical scale in 1872, when Eckman and his associates first put the present Eckman process into operation. It was introduced into England in 1884, and although it is practically a new process, it has developed very rapidly, being worked to a considerable extent throughout this country. The work of Mitscherlich in Germany, and Partington in America, aided largely in its rapid development.

In the sulphite process the lignin in the wood is decomposed by the bisulphites of calcium and magnesium into sugar and calcium and magnesium salts of the dibasic lignin-sulphonic acid. Coniferin is decomposed in an analogous manner into coniferin-sulphonic acid. If there is not sufficient lime present to neutralize the lignin-sulphonic acid formed, the lignin glycide suffers polymerization and is converted into a dark-brown resin that is insoluble in sulphurous acid.

45. Bisulphite Liquor.—At the present time a solution of bisulphite of lime and magnesia, with excess of SO_2, is almost exclusively used in this process, although a solution of bisulphite of soda is used to some extent, which solution produces a soft, white pulp. This liquor is prepared by several different methods, a few of which are described as follows:

46. Sulphurous acid gas, or sulphur dioxide, is almost invariably prepared from sulphur. This is accomplished in a set of sulphur burners, similar to the one shown in Fig. 16. The sulphur is shoveled into the burner at the doorway *a* and is spread out in a thin layer over the floor, after which it is ignited. It will support its own combustion, and just enough air should be admitted to properly burn the sulphur to sulphur dioxide SO_2, which requires 53.81 cubic feet per pound of sulphur. The amount of air used is regulated by the amount of draft admitted to the burner. The door *b* is suspended on a chain *c*, which runs over a pulley *d*, and has a weight *e* attached to the other end, by which means the door can be easily moved up or down in order to regulate the draft. The burner is kept cool by water running on the top of it from a pipe *f* and escaping at *h*, there being about an inch of water on the top continually. The SO_2 gas formed according to the reaction $S + O_2 = SO_2$ is conducted through the pipe *g* to the coolers. The burner should be made air-tight all over,

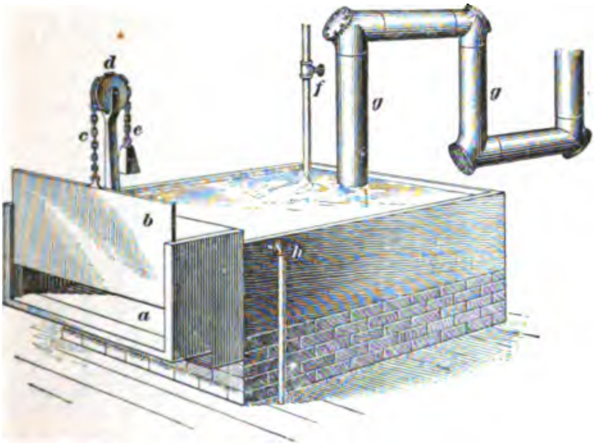


FIG 16

excepting where the air supply is regulated, and there should be an opening under the burner extending its full length, in order to keep it as cool as possible. If too much air gets into the burner there is a tendency to form sulphur trioxide and also to overheating, and, consequently, to sublimation of sulphur.

In case of copper or iron pyrites being used, which is the case in some foreign mills and in one or two mills in this country, the mineral is crushed and roasted on the grate bars of some form of pyrite burner and the gas conducted to the cooler as above. The chief objection to the use of pryites is the difficulty found in removing the fine dust, which is carried along with the burner gas and eventually finds its way into the bisulphite liquor.

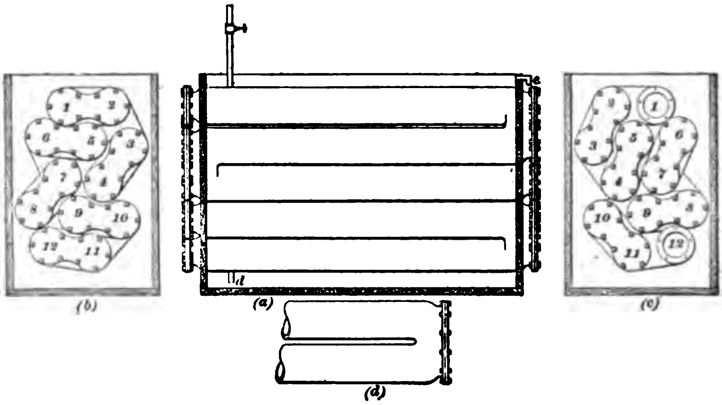


FIG. 17

It is always best to pass the gas through a series of large pipes that extend up and down (see *g*, Fig. 16). These pipes should not have any abrupt turns in them. They may be made of iron, and where it is necessary to turn, crosses should be used, which are provided with removable caps so that the sublimed sulphur can be removed from time to time. The object of this piping is to catch any sublimed sulphur coming from the burner, and thus preventing its entering and clogging up the cooler. The gas is then passed through the cooler, which consists of a coil of lead piping encased in a wood tank filled with water, and has a continual stream of cold water entering and leaving it.

47. In Fig. 17 (*a*), (*b*), (*c*), and (*d*) is shown the construction of one of these coolers. A side view of the interior piping is shown in (*a*). The box is kept full of water, which is constantly flowing in at the bottom through the pipe *d*, and leaving the box through the pipe *e*. The two end views (*b*) and (*c*) show how the pipes are arranged in the cooler. The gas enters from the up-and-down pipes, passing in through *1*, returning through *2*, back again through *3*, returning through *4*, etc., and finally passing out through *12*. Caps are bolted over the ends of the pipes, as shown in (*d*), so that they can be removed and the pipes cleaned out if they become stopped up. The pipes are made of lead.

One of these coolers is provided for each sulphur burner, but the exit pipes of two coolers unite in one main for each set of absorption tanks. After leaving the cooler, the gas then passes to the absorption tanks, where the bisulphite liquor is formed.

48. Methods of Absorption of Sulphur Dioxide.—There are a number of forms of absorption apparatus, some of which adhere to the old form of a lime tower, in which the lime is placed in lumps and the gas drawn up through the tower, while water is sprinkling down over the lime. The solution drawn off at the bottom is the bisulphite liquor. Milk of lime is most commonly used at the present time for the formation of bisulphite liquors.

49. McDougald Absorption Apparatus.—This consists of three tight tanks, fitted with agitators, which tanks are nearly filled with milk of lime. The gas from the cooler enters the first tank near the bottom, passes up through the lime water, and out at the top, thence to the bottom of the second tank, and so on through the series. The tanks are all so connected that the milk of lime can be transferred from one to the other. When the first tank is up to strength, and is drawn off into the settling tank, the valves between the tanks are opened, and fresh milk of lime run into the last tank, until they are all at the same level, when the valves are again closed. In the McDougald process the sulphur is burned under pressure and the gas thereby forced through the cooler, tanks, etc., which is a disadvantage, owing to the fact that the combustion proceeds more rapidly and a large proportion of SO_3 is formed.

50. A modification of this apparatus that furnishes good results is one in which the tanks are placed one above the other and the gas carried through the series by means of a vacuum pump attached to the top one. The tanks are all provided with agitators and are air-tight. The milk of lime is pumped up into a tank above the series of absorption tanks, where it is kept mixed by means of an agitator. This lime water is run into the top absorption tank near the bottom, and flows from an outlet near the top, entering the next lower one, near the bottom, and so on. At the same time the gas is being drawn up through the tanks, the pipe entering at the top of the bottom tank but discharging the gas at the bottom. The gas then passes up through the milk of lime and is conducted by a pipe from the top of the bottom tank into the top of the next higher tank, discharging at the bottom, and so on through the series. By this arrangement there is a continual flow of liquor through the apparatus. Fresh milk of lime is continually entering the top tank, while a corresponding amount of finished liquor is being drawn from the bottom tank.

51. Burgess Absorption Apparatus.—A very effective absorption apparatus for the manufacture of bisulphite liquor is the Burgess triple acid-absorption tank, shown in Fig. 18 (*a*), (*b*), and (*c*). Fig. 18 (*b*) represents a vertical section, Fig. 18 (*c*) an elevation, and Fig. 18 (*a*) a section through the top compartment, showing the hollow arms *h*, *h*, *h'*, *h'*. The lime water is fed into the top compartment of the tank at *c*, Fig. 18 (*b*), and flows into the middle compartment through the overflow pipe *d*, and from the middle to the bottom compartment through the overflow pipe *e*. A pipe from the vacuum pump is connected at *a*, which draws the sulphur dioxide from the sulphur ovens through the gas

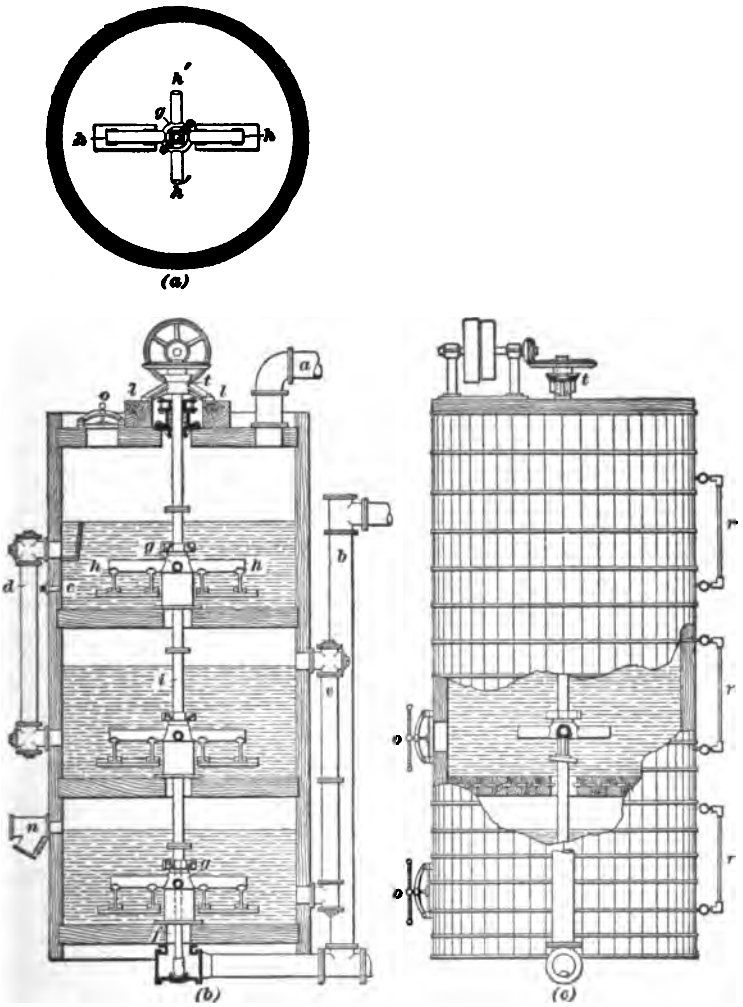

h'
g
h
h
h
(a)
o
l
t
l
a
g
h
h
d
c
b
i
e
n
g
(b)
t
r
o
r
o
r
(c)

FIG. 18

cooler to pipe *b*, which passes to the bottom and enters the tank at the center. The gas is drawn through pipe *b* and sleeve *f* into the dome *g*, and thence through the hollow arms *h*, *h*, *h'*, *h'* into the lime water, as shown in Fig. 18 (*a*). The gas passing up through the lime water in the bottom compartment is drawn by the vacuum through the sleeve and hollow arms into the middle compartment, and the same operation is again repeated in the top compartment. Wooden agitators are attached to each hollow arm *h*, *h*, and throw the liquor from the bottom up into the gas coming from the ends of the hollow arms, thus saving some of the work of the vacuum pump. The center shaft *i* revolves, thus distributing the gas as it enters the three compartments. This shaft is supported at the top by the bridge truck, resting on the beams *l*, *l*, and runs on ball bearings *t*. There is no weight on the other end, but merely a guide for the shaft. The tank is made of wood, while the piping, shaft, and agitator are made of bronze.

52. The finished product, calcium and magnesium bisulphites, in the bottom compartment, is drawn off to the storage tank by means of the **Y** valve *n*. Indicator glasses *r* are attached to each compartment, showing the condition of the liquor in each. There is a manhole *o* in each compartment. The tanks are made in various sizes, each tank making acid for from 25 to 100 tons of pulp per day.

The above absorption apparatus is very simple and compact, requiring very little labor to operate it and occupying very little space. There is only one tank to be kept airtight, while in the other systems mentioned there are three of them and sometimes four.

The lime used in all of the above operations is magnesia lime, well burned, which is first slaked in an iron tank provided with an agitator, after which it is run into a large one, diluted to the strength required, allowed to cool, and pumped up into the tank above, for use in the absorption apparatus. The liquor is pumped up into the storage tank, and in order to get clear liquor for the digesters, the delivery pipe should be attached to a float, so that the liquor is always drawn from the top.

The bisulphites of calcium and magnesium are formed according to the following equations; first the monosulphites are formed, and each molecule of these taking on another molecule of sulphurous acid, is converted into the bisulphites, as shown by the following equations:

$$(1)\quad Ca(OH_2) + Mg(OH)_2 + 2H_2SO_3 = CaSO_3 + MgSO_3 + 2H_2O$$

$$(2)\quad CaSO_3 + MgSO_3 + 2H_2SO_3 = CaH_2S_2O_6 + MgH_2S_2O_6$$

53. Preparation of the Wood.—In the sulphite process, knots, pieces of bark, and decayed wood are hardly acted on by the bisulphite liquor, and should therefore be removed before sending the chips to the digester. This is done in some mills by boring out the knots and cutting out the rotten parts, while in other mills, when slabs are used, the slabs are sent to a knotting mill, where the knots are cut out by means of revolving circular saws. In some foreign countries, where labor is cheap, the chips from the chipper are thrown on an endless belt, where the knots are picked out by children as the chips pass.

The chipper used in the preparation of the wood is similar to the one used in the preparation of wood for the soda process. The chips are passed through a long screen in order to remove the sawdust, dirt, etc. A convenient form of screen, and one that furnishes chips of a uniform size, consists of a double revolving cylinder. The chips from the chipper enter the inner cylinder, the larger pieces passing on to the other end while the smaller ones drop through the openings in the cylinder into the interior of the outer cylinder, which is covered with a coarse-mesh wire cloth, through which the sawdust and dirt pass, while the chips of the required size pass through to the other end of the cylinder, where they are caught and conveyed to the top floor of the digester room, by means of an endless chain of buckets. The wood generally used is spruce, although hemlock, poplar, and fir are used to some extent.

54. Sulphite Digesters.—The sulphite digesters vary in form, the same as the soda-pulp digesters; but aside from this they must have a lining that will resist the action of acids. Pure lead, owing to the formation of insoluble lead sulphate, which coats the surface, furnishes a good acid-resisting material, but lead containing small quantities of antimony and copper is still better for this purpose. On account of the unequal expansion of lead and iron when heated, and the failure of the lead to assume its original condition when cooled, the lead linings have a tendency to creep, so that they have given place almost entirely to other materials for linings. A very good lining, which still makes use of lead as one of the materials, is the " non-antem " sulphite digester lining, which is made of the following materials: lead, cement, and brick, given in order of their position from the shell to the interior, and consist of ½-inch, ¾-inch, and 9-inch layers, respectively.

The interior of some digesters are bricked and lined with a mixture of various cements, while others are cemented on the inside and then faced with a layer of tiles; and, in fact, a great variety of materials are now used for linings, such as sulphite of lime, double silicates of iron and lime, glass, etc.

55. After every cook the digester should be thoroughly examined, and if there are any loose places in the lining they should be repaired before putting on another cook.

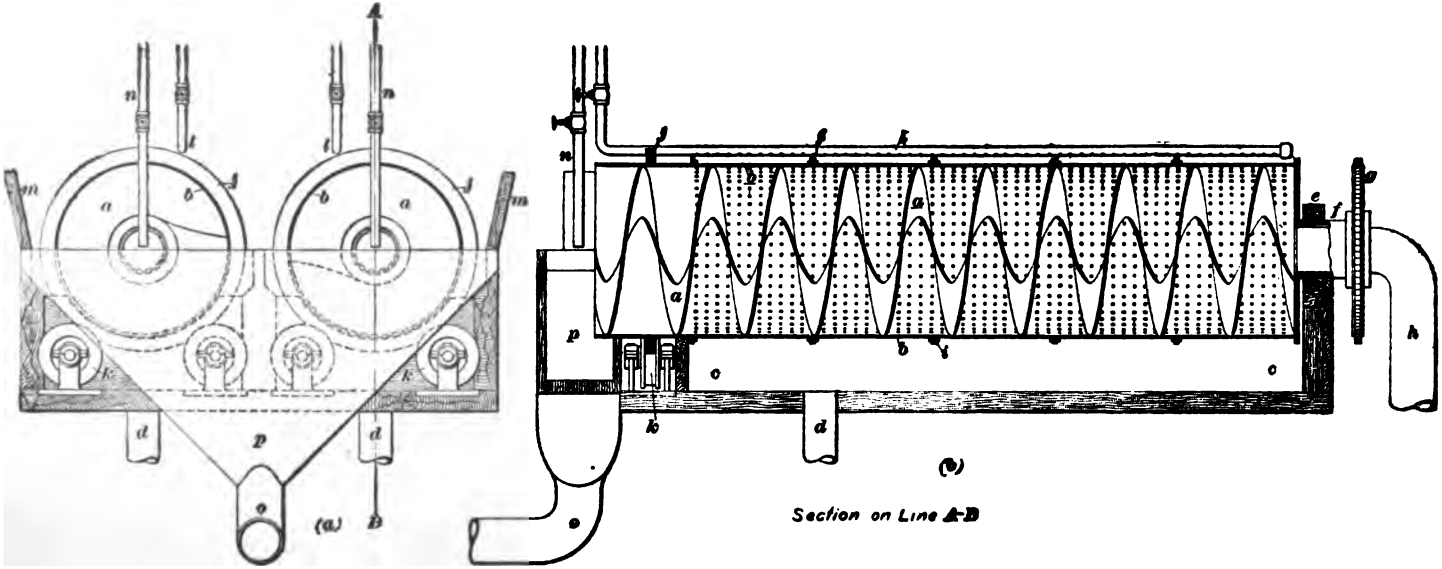
Some paper makers are of the opinion that better results can be obtained by the use of rotary digesters, quite a number of which are in use at the present time. They claim that there is less danger of making black chips and that less liquor is used, but it is the most general opinion that a better quality of pulp is produced in the upright digesters.

The digesters vary in size, some of the upright ones being 50 feet by 15 feet in diameter by 50 feet in length, and holding from 28 to 30 cords of wood. Those in most common use are from 12 to 14 feet in diameter and from 36 to 40 feet in length.

56. Charging and Cooking.—The chips and liquor are charged at the top, the digester being filled as full as possible with chips, which will settle down when the steam is turned on. The liquor is run through a large pipe in order to get it in as quickly as possible, the strength of which is generally from 6° to 7° Baumé at 60° F., and contains about 3.5 per cent of sulphurous acid.

The pressure should not be put on too rapidly, as it will have a tendency to burn the chips, if they are not sufficiently soaked with liquor. The pressure varies from 45 to 85 pounds. There is usually a thermometer arranged on the side of the digester, whereby the temperature on the inside can be read at any time. The cooking is generally carried on at a temperature of about 300° F. On account of the large amount of gas generated, the temperature is a better guide to go by than the pressure. It is customary to blow the gas off at the top of the digester from time to time, near the end of the cook. The blow-off gas may be recovered by sending it through a lead pipe to an absorption tank. The lead pipe passing through a tower is cooled by water and in this way the gas is freed from steam and the latter condensed. Many other equally effective methods have been proposed and are used in different works. By carefully utilizing the recovered gas, 100 pounds of pulp can be produced with 10 to 11 pounds of sulphur. Some of the digesters are provided with a circulating device, as in the soda digesters, by which the liquor is carried from the bottom, under a perforated plate, up to the top, where it is discharged on top of the wood, keeping up a continuous circulation.

The time required for each cook depends on the wood used, the strength of liquor, and the pressure. Boiling with a liquor containing 3.5 per cent. sulphurous acid at a pressure of 75 pounds, it requires from 16 to 20 hours. It requires from 1,000 to 1,200 gallons of this liquor for each cord of wood cooked. When the cooking is complete, the pressure is blown down to about 30 pounds, and the pulp discharged from the digester (this should be done as quickly as possible, or the heat in the digester, in the absence of sulphite of lime, will turn the pulp brown) into a large draining tank, where it is washed to some extent by running water


A
n
t
b
j
a
m
k
d
p
o
(a)
B
g
k
e
f
h
c
i
(b)

Section on Line A-B

FIG. 19

upon it. It is then pumped up to the washers, where it undergoes a further washing.

57. Worm Washer.—A very convenient form of washer, known as the worm washer, is shown in Fig. 19 (*a*) and (*b*). Fig. 19 (*a*) is an end elevation of the end where the washed stock is delivered, and Fig. 19 (*b*) is a longitudinal section on the line *A B* of Fig. 19 (*a*). It consists of a copper worm *a*, somewhat resembling an auger, enclosed in a perforated copper cylinder *b*. The cylinder is partly enclosed by a box *c*, which catches the wash water and conveys it away through the pipe *d*. There is a support *e* at one end of the box, through which the hollow trunnion *f* of the cylinder passes. There is a large cog *g* at the end of the trunnion, and an endless chain connects the cogs of the two cylinders together, and they are made to revolve at the same time. The stock enters the cylinder through the pipe *h* that passes through the hollow trunnion, which trunnion revolves round the pipe. There are heavy iron bands *i* encircling the cylinder at intervals, and at the other end it is encircled by a track *j*, which passes through a hollow wheel *k*, and thus holds the cylinder steady while revolving.

There is a perforated water pipe *l* extending along the whole length of the cylinder, which forcibly emits small jets of water against it. This water penetrates the cylinder and washes the pulp during its passage through the worm. A dashboard *m* extends from the box about as high as the cylinder, and thus prevents the water from splashing out.

The cylinder is perforated and the jets are emitted only on the surface, which is directly over the box. At the end of the cylinder there is a funnel shaped box *p*, into which the washed stock from two cylinders is deposited and washed down by water from the pipes *n* into the exit pipe *o*. There is one of the pipes *n* at each side of the box *p*. The waste-water box *c* is hollowed out at this end, to allow the drums to pass through.

In its passage through the worm the stock is turned over and over, thus exposing all surfaces to the washing action.

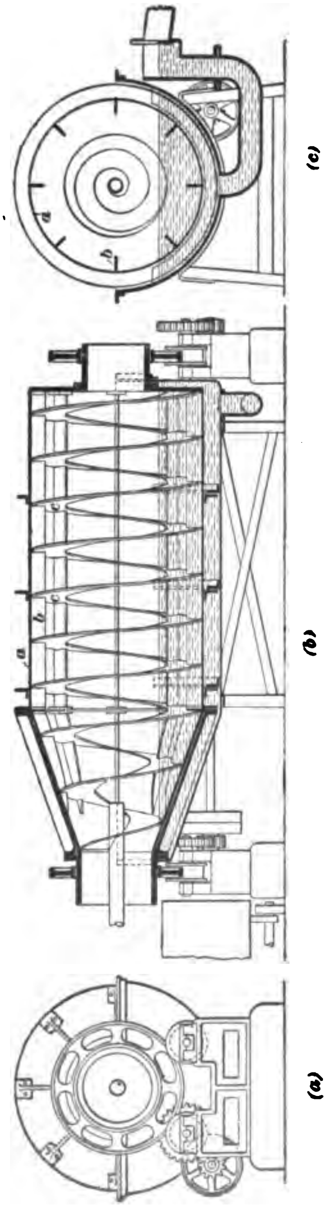


FIG. 20

The accompanying illustration taken from the "Paper Trade Journal" shows an apparatus for washing pulp that is somewhat similar to the worm washer described above. Fig. 20 (*b*) is a longitudinal section, Fig. 20 (*c*) is a cross-section, and Fig. 20 (*a*) is an end elevation. The vat, or trough, is mounted upon a suitable support and receives the drum arranged horizontally, having end trunnions journaled in suitable bearings. The drum consists of a perforated shell *a*, which has a series of ribs *b* projecting inward from its side, at certain distances apart. A perforated blade *c* is wound helically around the inner periphery of the shell and extends from end to end. The water is admitted through a pipe at the conical end of the drum, the cleansed fiber being also delivered at this end through a central opening in the trunnion. The unwashed fiber is admitted through a central opening in the trunnion at the opposite end of the drum. In the bottom of the vat is formed a transverse, vertical web, to prevent the direct passage of the clean water towards the outlet. By the rotation of the drum and helix the fibrous material is gradually carried along its whole length and through the washing water, the level of which is kept above the helical blade to insure the immersion of the fiber carried along. During the rotation of the drum the fibrous material is also repeatedly lifted and turned over. When the fiber reaches the conical end it is raised up clear of the water.

58. The excess of water in this pulp is usually removed by drum washers in the bleachers, the stock being pumped from the screens directly to the bleachers. In the sulphite process, unlike the soda process, the resins are not converted into soluble soaps, only the soluble parts of the resins going over with the sulphite lye, and greater care should be exercised in washing out the dissolved resin adhering with the lye to the fiber before it hardens again. There are some forms of resin that are insoluble in the hot sulphite lye and, therefore, stay in the pulp, causing brown and yellowish spots to appear in the paper. The particles of resin, being lighter than water, float on the surface, and many paper makers use laths covered with strips of long-haired felts, which float on the thin pulp when passing over the sand traps in the beaters and retain most of the resin.

59. Disposition of Waste Sulphite Liquor.—Quite a little investigation of the waste liquor from the sulphite process has been carried on, and it has been found that it can be worked up and used as a sizing agent by precipitating with sulphate of aluminum, but it is not used as yet on a practical scale.

The waste sulphite liquor has also been evaporated, worked up with coal dust into briquets, and used as fuel.

60. Ground Plan of a Typical Sulphite Pulp Mill. In Fig. 21 is shown a ground plan of a typical sulphite pulp mill.

A is the sulphur storeroom.

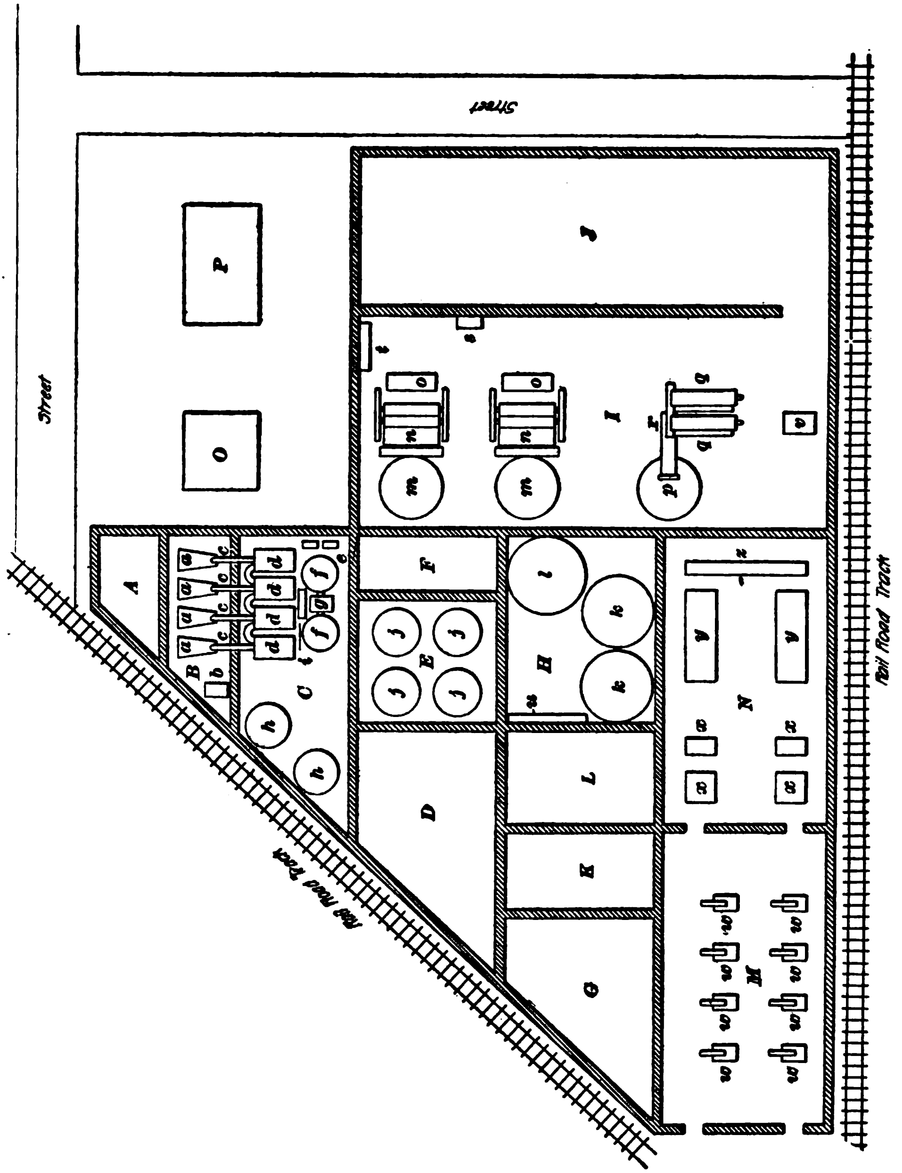

Street
Street
Rail Road Track
Rail Road Track
A
B
C
D
E
F
G
H
I
J
K
L
M
N
O
P
a
b
c
d
e
f
g
h
j
k
l
m
n
o
p
q
r
s
t
u
v
w
x
y
z

FIG. 21

B is the sulphur-burner room; *a*, the sulphur burners; *b*, an elevator for conveying lime up to the lime storeroom above the mixers.

C is the absorption room; *c*, the up-and-down preliminary cooling pipes; *d*, the coolers; *e*, the vacuum pumps; *f*, the lower absorption tanks, the highest set of absorption tanks being directly over the coolers; *g*, the tank into which the finished bisulphite liquor runs; *h*, the lime mixers; and *i*, the stairs leading up to the absorption tanks.

D is the boiler room.

E is the acid and liquor storage room; *j*, the tanks in which bisulphite liquor and sulphurous-acid solution used in bleaching are stored.

F is the engine room.

G is the supply room.

H is the digester room; *k*, the digesters; *l*, the tank in which the cooked stock is given a preliminary washing; and *u*, the stairs leading up to the top floor of the digester room.

I is the wet-machine room; *m*, chests of bleached stock for wet machines; *n*, the screens and wet machines; *o*, the tables on which the pulp is folded; *p*, the chest into which the washed stock passes before being pumped up stairs to the screens and bleachers; *q*, the worm washers; *r*, the cylinder through which the stock passes after coming from the washers, which gives it a thorough breaking up; *s*, the scales; *t*, the stairs leading to the upper floor; and *v*, the elevator leading to the upper floor.

The screens and bleachers are on the second floor of the wet-machine room.

J is the pulp storeroom.

K is the pipe shop.

L is the engine room.

M is the knotting department; and *w*, the saws.

N is the chipper room; *x*, the chippers; *y*, the screens; and *z*, the conveyer that takes the chips up to the top of the digesters.

O is the laboratory.

P is the office.

OTHER PROCESSES FOR TREATING WOOD

61. Sulphate Process.—Sodium sulphate is used to some extent in cooking wood and produces a pulp of excellent quality. It is mixed with one-third its weight of caustic soda. The density of liquor used varies from 6° to 13° Baumé. The time required depends on the strength of liquor used and the pressure at which it is cooked, varying from 30 to 40 hours. The pressure varies from 75 to 150 pounds. The coniferous woods are exclusively used as raw materials.

The soda compounds are recovered as in the soda process, the liquor being evaporated and calcined, yielding a reddish-brown ash. There is a loss of 10 to 20 per cent. in the recovery, which is made up by addition of fresh sulphate and the whole heated with 20 to 25 per cent. of lime. The main objection to this process is the formation of organic sulphur compounds of objectionable and penetrating odor. Another objection is the length of time required to cook the wood.

62. Pictet and Brélaz's Process.—The wood is subjected to the action of a supersaturated solution of sulphurous acid, under a vacuum, at a temperature not exceeding 212° F. The liquor permeates the wood, dissolving out all the cementing constituents which envelop the fibers. The digesters are lined with lead. It requires from 12 to 24 hours for complete disintegration, according to the nature of the wood used. This pulp is readily bleached with chloride of lime.

63. Barre and Bondel's Nitric-Acid Process.—The wood is digested for 24 hours in a 50-per-cent. solution of cold nitric acid. It is washed first with hot water and finally with a weak solution of sodium carbonate.

64. Nitrohydrochloric-Acid Process. — There have been several processes patented for disintegrating wood by the use of various mixtures of nitric and hydrochloric acids, all of which use more hydrochloric than nitric, the strength of the mixture varying according to the temperature at which the disintegration takes place. When the mixture is used cold, it is made of strong acids, but when used hot, the acids are diluted about twenty times with water.

The main difficulties encountered with the use of acids are as follows: The principal one is in being able to provide vessels that will resist the powerful corrosive action.

Nitric acid will form an explosive substance of the guncotton series, and there is, therefore, a great risk involved in drying the pulp obtained by a process in which this acid is used.

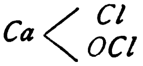
The objection to using the ordinary mineral acids, hydrochloric and sulphuric, as well as sulphurous acids, is as follows: When they act on wood at moderately high temperatures, the decomposition products accumulate very rapidly and undergo a secondary decomposition, tending towards the formation of dark-colored tarry matter, which prevents the formation of pure cellulose. This is overcome in the sulphite process by introducing a base, which unites with the organic acids formed and produces soluble salts of these acids.

BLEACHING THE VARIOUS FIBERS

A

65. General Remarks.—After washing the various fibers, they are always found to be more or less colored, owing to the fact that a portion of the non-cellulose constituents survive the foregoing processes. The next operation will be to remove this coloring matter as much as possible, and thereby produce pure white fiber, for the manufacture of paper. This is accomplished by the bleaching process.

Bleaching is simply an oxidizing action; and although there are other agents to be had, chlorine or compounds of chlorine are principally used to accomplish this purpose. Of these compounds of chlorine, bleaching powder, made by passing chlorine gas through slaked lime, is the one most used in the paper industry, the formula of which is generally accepted as



$$Ca\left\langle\begin{matrix}Cl\\OCl\end{matrix}\right.$$

Some authorities advocate the theory that the bleaching is accomplished as follows: The bleaching powder, when treated with water, is resolved into equal molecules of calcium chloride $CaCl_2$ and calcium hypochlorite $Ca(OCl)_2$, the latter, which is the active bleaching agent, splitting up into $CaCl_2 + O_2$. It is, however, most generally accepted that the bleaching action is due to the liberated chlorine combining with the hydrogen of the water, setting free oxygen, which does the work. The bleaching powder should be stored in a cool, dry place, otherwise it will lose strength; and when mixed for use, it should be used up as quickly as possible.

66. Preparation of the Solution of Bleaching Powder.—The tanks used are generally made of iron, and are provided with agitators and siphon pipes, similar to those used in causticizing pans. They vary in size, holding from 1,000 to 2,500 gallons, the most convenient size being one in which either one or two whole casks of bleaching powder can be used, and the bleach made up to the required strength, thus avoiding the necessity of splitting casks. The water used in mixing the bleach should not exceed 70° F., and it is better, provided the bleach settles well, to keep it as near 60° F. as possible.

The tank is filled about two-thirds full with water, the agitators started, and the bleaching powder dumped in. The agitation is continued until all the lumps are well broken up, after which it is stopped and the bleach allowed to settle. In some mills, just enough bleach is used to make the liquor up to the strength required, the washes being used for making up other tanks and washes, the final wash being made with water. The washes are made similar to those of the lime sludge in the manufacture of caustic-soda liquor. The method most generally adopted is to make the bleach up stronger than desired and mix in a tank in the cellar with washes of other tanks, until the required strength is reached. Sufficient washes should be made until practically all the chlorine has been washed out of the sludge, allowing the bleach to settle after each wash. The balance of these washes, after the strong bleach is diluted to the required strength, are run into a separate tank and used in place of water for making up strong bleach.

67. The percentage of available chlorine is not the only factor to consider when buying bleach, as the settling quality figures to a considerable extent. A bleach that is high in available chlorine, but a poor settler, is of no more value to a concern that has a limited capacity for making liquor than one that has less chlorine, but settles well. It is due to this fact that the manufacturers of bleaching powders in this country are unable to keep the foreign bleach out of our market. It is advisable that only clear liquor be used in bleaching.

The sludge from bleaching powder, consisting principally of calcium hydrate and calcium carbonate, is washed out into the sewer after the last wash has been run off.

Sulphites are oxidized to sulphates by the action of hypochlorites

$$2Na_2SO_3 + Ca(OCl)_2 = 2Na_2SO_4 + CaCl_2,$$

hence, they are useful in neutralizing excess of bleach.

68. Quite a number of mills, as has been stated, do their bleaching in the breakers; but it is generally customary to transfer the washed pulp, with the necessary amount of water, to what are termed *potchers*. These potchers are of various forms, some of which are provided with steam pipes to heat up the pulp while bleaching. Some are provided with drum washers and all are provided with agitators or some means of keeping the pulp in circulation during the bleaching operation.

69. Bleaching Rags.—Pulp from rags is generally bleached in revolving barrels made of wood, lined with a suitable acid-resisting material. The rags are put into the barrel through a manhole, the required amount of bleaching liquor added, and the barrel set in motion. The bleaching of rags is better accomplished by the addition of either hydrochloric or sulphuric acids during the process of bleaching. The acid should be very much diluted when added, and the addition made after the bleach has been acting on the pulp for some time. When acid is added to accelerate the bleaching action, the process is termed *acid bleaching*. Acetic acid has been used to a considerable extent to assist the action of bleach liquor in bleaching rags, which only requires the addition of a small quantity, from the fact that during the operation the acid is regenerated according to the following equations:

$$\text{(1)} \quad Ca(OCl)_2 + 2CH_3CO_2H = (CH_3CO_2)_2Ca + 2HOCl$$

$$\text{(2)} \quad 2HOCl = 2HCl + O_2$$

$$\text{(3)} \quad (CH_3CO_2)_2Ca + 2HCl = 2CH_3CO_2H + CaCl_2$$

The amount of bleach necessary to produce a good color depends on the thoroughness of previous treatments, but may be given as 2 to 5 pounds of bleaching powder to every 100 pounds of pulp.

70. Bleaching Esparto.—This fiber is very often bleached in the washing and breaking engine, where it is subjected to an acid bleaching. The required quantity of bleach liquor is added, and after mixing for about ½ hour, the highly diluted acid is added (using about 6 ounces of acid to 100 pounds of fiber) and the bleaching continued until a good color is produced. The liquor used tests about 4° Baumé at 60° F. This fiber is also bleached in large potchers made of brick, lined with cement, the agitation being accomplished by means of large revolving paddles made of either wood or iron, preferably the latter. From 10 to 15 pounds of bleaching powder is necessary to bring 100 pounds of esparto to a good color.

71. Bleaching Straw.—This fiber is bleached by methods similar to those used for bleaching esparto. The amount of bleach required is from 8 to 12 pounds for each 100 pounds of pulp.

72. Bleaching Jute and Manila.—Jute and manila are usually bleached in the washing engine, and the chloride of lime, when used, is added in a very weak solution, which bleaches the fiber to a cream color, oxidizing it to some extent. Strong bleach should not be used, as it will badly chlorinate the fiber. It is better to use a weak solution of sodium hypochlorite in bleaching jute, which will prevent the formation of the chlorinated compound. As it is difficult to bleach these fibers to a white, they are generally used in papers that do not require a high color. From 9 to 10 pounds of bleaching powder is necessary to each 100 pounds of pulp.

73. Bleaching Ground Wood.—Owing to the fact that ground wood contains nearly all the intercellular constituents of the wood, which have to be removed by the bleach before any action takes place upon the coloring matter of the fiber, it cannot be economically bleached, and is, therefore, used in common papers, as before stated.

74. Bleaching Wood Fiber.—There are a great variety of forms of bleaching potchers for bleaching wood fiber. Some very much resemble a beating engine (in fact, some paper makers bleach their stock in the "beaters"), others are large cylindrical wooden tanks, provided with an agitator in the center, which consists of a central rod with paddles attached at different heights, while still others are large, open, tile-lined vats made of brick, having wings attached to a revolving horizontal shaft, which keeps the stock agitated during the bleaching operation. It has been found by practice that better results can be obtained by bleaching the stock in open bleachers. All forms of bleachers have a steam line running into them, by which the stock can be heated during the operation. This must be done very cautiously, as there is great danger of chlorinating the fiber if heated too highly. There is also danger of heating it too highly in one spot (where the steam enters), with the same result. Better results can be obtained by heating the stock up to the required temperature before adding the bleaching liquor. It is advisable to keep the temperature under 115° F., otherwise there will be trouble from time to time with chlorinated fiber.

75. There are times when difficulty arises in bringing a bleacher of stock up to color, which is generally due to insufficient treatment in previous operations. In such a case the action can be greatly assisted by washing out the products of the bleaching action, treating with a weak solution of alkali and washing again. After this treatment, the most refractory pulp can be brought up to color by again treating with bleaching solution. In bleaching sulphite pulp, the fact that 14 to 22 per cent. of bleaching powder is required indicates that it has to perform other actions besides bleaching. The amount of bleaching powder required increases with the amount of incrusting matter left in the pulp. The use of the soda solution above mentioned before bleaching will greatly reduce the bleach figure.

76. In bleaching sulphite pulp, some paper makers warm the pulp in a 13-per-cent. solution of bleaching powder, and after an hour's time add a 2.5-per-cent. solution of sulphuric acid. The pulp is then washed for 2½ hours and rebleached with a 2-per-cent. solution of bleaching powder, finally adding a ½-per-cent. solution of sulphuric acid.

In bleaching the soda or sulphite fiber, the strength of the bleach solution used is generally from 3½° to 4° Baumé at 60° F. (about ½ pound of bleaching powder to the gallon). It is advisable to keep it as regular as possible in order that the results obtained will be uniform. The bleaching of wood fiber requires from 12 to 25 pounds of bleaching powder per 100 pounds of pulp, depending on the wood used, the process by which the fiber was isolated, etc. After bleaching any fiber, it must be well washed in order to remove excess of bleach and soluble by-products. This is done in some mills by means of a drum washer in the potchers; in others by the same operation in the beaters, while in still others the bleached stock is pumped with a large quantity of fresh water to large drainers or chests, which have perforated bottoms, and allowed to stand until it drains down solid, after which it is pumped with a large amount of water to the beaters, mixers, or pulp-machine stuff chest.

77. It is generally customary to use some form of antichlor to neutralize last traces of bleach after the stock has come up to the required color, sulphite and hyposulphite of soda being used to a great extent. Sulphurous acid is also used to some extent, which removes the slight yellow tint left in pulp after bleaching, bringing it to a fine white. On exposure to the atmosphere for any length of time, this yellow tint will appear again, owing to the coloring matter that was temporarily removed by the reducing action of the sulphurous acid, being again oxidized. When treated with an antichlor, the pulp must also be again washed as above.

78. Bleaching powder, as before stated, is not the only bleaching agent that can be used, as sodium hypochlorite, aluminum hypochlorite, magnesium hypochlorite, and zinc hypochlorite have been used from time to time, but owing to their greater cost they have not been worked on a commercial scale.

79. Electrolytic Bleaching.—There has been considerable experimenting during the past few years upon methods of preparing bleach liquor by the electrolysis of common salt, and as a result of these experiments such a degree of proficiency has been gained that electrolytic bleach plants are now being introduced into some of the mills in this country.

The process of bleaching with the product of the electrolysis of an alkaline chloride was first worked on a commercial basis in 1886, when M. Hermite developed the process for the production of magnesium hypochlorite by the electrolysis of a 5-per-cent. solution of magnesium chloride.

The process of electrolytic bleaching is based on certain well-known principles of electricity, a general idea of which is here given.

A metallic conductor does not suffer any apparent change when a current of electricity passes through it, but various magnetic and heating effects are produced. Some liquids, as well as solids, are good insulators, while others conduct electricity, and are termed *electrolytes.* The latter suffer decomposition in proportion to the amount of current passing through them. The poles are the points at which the current enters and leaves the liquid, the former being termed the anode and the latter the cathode. The products of decomposition of the liquids are observed at the poles, and are termed the *anion* at the anode and cation at the cathode. In the process of decomposition of fused common salt, chlorine is given off at the anode and sodium at the cathode. When a solution of common salt is used, a secondary reaction takes place, due to contact of the liberated ions, and there is a tendency towards the formation of sodium hypochlorite, which remains in solution, and hydrogen, which escapes at the cathode. There is also a decomposition of the water itself into hydrogen and oxygen, and the oxygen that is liberated at the anode will attack the material of which it is made, and, in the case of carbon, destroy it in a short time. It is the aim of the inventor to procure an anode that will resist the action of the products of electrolysis, platinum being the best in this respect, but as this material is so expensive, different forms of carbon have been used to a considerable extent. It is also advantageous to use as little water as possible, so that nearly all inventors use a saturated brine solution. The quantity of electrolyte decomposed by the passage through it of a given quantity of electricity is always the same. The current efficiency of a cell is determined by dividing the quantity found by the theoretical amount.

80. That which causes electricity to flow from a point of high potential to a point of low potential is called the **electromotive force** (E. M. F.), the unit of which is the **volt.** The unit of quantity of current is the **coulomb;** the unit of rate of flow, which is 1 coulomb per second, is called the **ampere;** the unit of resistance to the flow is the **ohm.** An E. M. F. of 1 volt will send a current of 1 ampere through a resistance of 1 ohm. A current of 1 ampere, theoretically, yields 1.34 grams of chlorine and

1.51 grams of caustic soda per hour. Owing to complications due to secondary reactions, in practice the yield is only about 1 gram of chlorine per ampere per hour. The power of a current in doing work is measured in units called **watts**.

A current of 1 ampere, under an E. M. F. of 1 volt, has an energy of 1 watt. One horsepower equals 746 watts.

Two pounds of coal is converted into 1 horsepower of mechanical energy, which, as stated, is equivalent to 746 watts. This is converted through the dynamo (with customary loss) into about 650 watts, which is the efficiency of the dynamo for each horsepower.

An E. M. F. of from 3 to 5 volts is required between the terminals, and since the power of a circuit in watts is equal to the number of amperes flowing, multiplied by the E. M. F. in volts, to produce 1,000 grams of chlorine it will require (assuming the E. M. F. to be 4 volts) 4,000 watts, or about 6 horsepower per hour, which means the consumption of 12 pounds of coal.

A great difficulty experienced in the process thus far set forth is in getting the caustic-soda solution free from salt, as it requires a diaphragm between the anode and cathode which will furnish as little resistance as possible and at the same time prevent the passage of brine solution through it. Several different materials are in use for diaphragms, such as unglazed earthernware, asbestos, etc.

81. A great many processes have been proposed for the production of chlorine and caustic soda by electrolysis, all of which have the same fundamental principles underlying them—working with a saturated brine solution, having a diaphragm between the poles, and separating the chlorine from the soda. There has, however, been considerable difference in the designs of the apparatus.

The dynamo generally used is one so wound as to deliver a continuous current of large volume under moderate voltage, say about 1,250 amperes at 120 volts. The cells are usually arranged in multiple arc, and the current is conducted to them through large copper conductors.

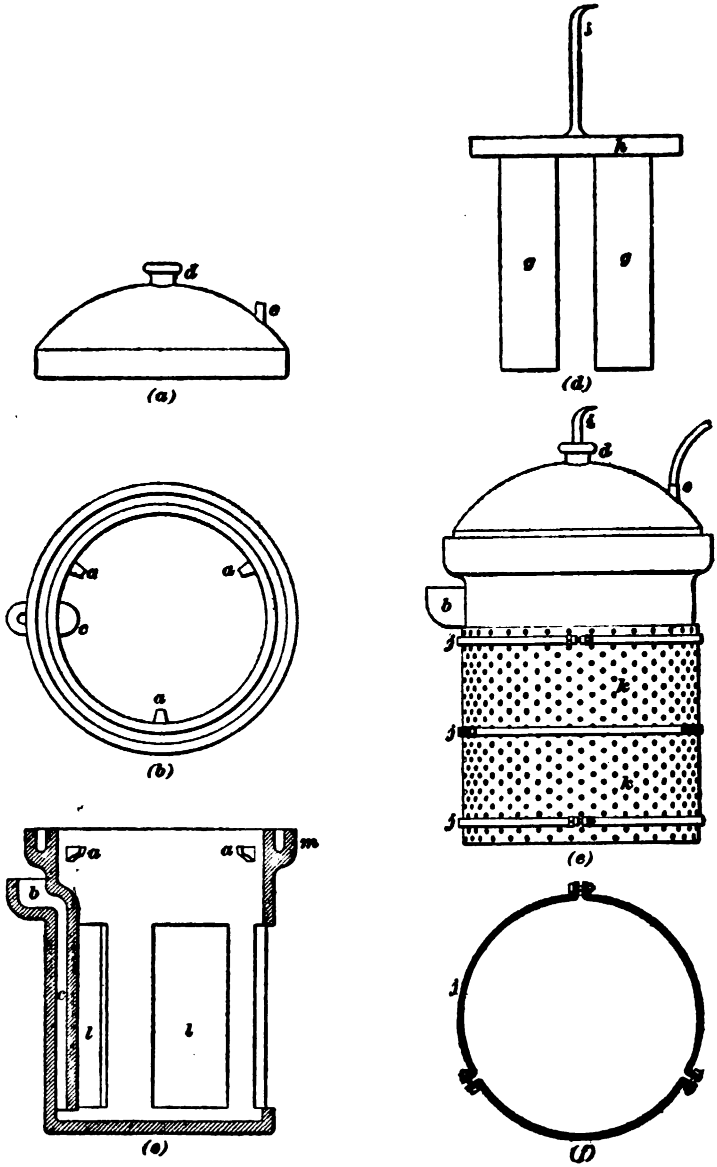

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FIG. 22

Following are descriptions of two styles of cells that have lately been patented and are now being worked on a paying basis at several paper mills.

82. The Mercer Cell.—This cell, shown in Fig. 22 (*a*), (*b*), (*c*), (*d*), (*e*), and (*f*), consists of an earthenware crock that is open at the top and sides (the side openings marked *l* are five in number) but closed at the bottom. There is a cup *b* at the side of the cell, into which the brine solution is poured, and it passes through the side of the cell into an inner tube *c*, which extends almost to the bottom of the cell, where the brine solution is discharged. This enables one to keep the cell full and to avoid the escape of gas while filling. The crock is enlarged at the top *m* and there is a raised inner ring, as shown, so that the lid (*a*) will fit down between the two rings and can be luted air-tight. The diaphragm used is asbestos, a sheet of which is wrapped around the openings in the crock, after which perforated sheet iron *k* is securely bound around the asbestos diaphragm by means of iron bands *j*, which are in three pieces and bolted together, as shown at Fig. 22 (*f*).

The anodes consist of round sticks of carbon *g*, four of which are attached to a lead support *h* that has a long lead projection *i* from its center. There are small projections *a* from the inside of the crock, upon which the lead support rests. The long lead projection passes out through the lid at *d* and is luted air-tight. It connects with the conductor from the dynamo. The chlorine gas escapes through *e* to the gas main. Six of these shells are placed in a long, oblong sheet-iron tank, which is just wide enough to admit them. The sheet-iron plate *k* touches the bottom of the tank, and this, together with the tank, forms the cathode. The tank is supported on pieces of glass. The soda passes through the diaphragm and unites with the water, which is continually flowing in at one end of the tank and out at the other, forming caustic-soda solution. The strength of the soda solution depends on the amount of water flowing into the tank. The solution is deep enough to cover the asbestos diaphragms. The chlorine gas passes through the gas main up the lime tower, where it comes in contact with the lime water. The lime water is mixed in a tank on the second floor, and passes down the tower to a tank below, where it is again pumped up into the tank above, this continuous circulation being kept up until the bleach solution has reached the required strength, when it is allowed to settle, the liquor run off, and the lime sludge, with the addition of more fresh lime, used again. This bleach liquor is tested from time to time for available chlorine by the usual arsenious acid tests in order to ascertain when it is up to the desired strength. The finished liquor should contain from 1.50 to 1.75 per cent. of available chlorine. If there is insufficient lime to take up all the chlorine to form hypochlorite, the excess of chlorine will cause the formation of chlorate, which results in the loss of the active bleaching agent; so that the Baumé test is not sufficient in testing the bleach liquor made by this process, and, as stated above, the arsenious acid test should be resorted to.

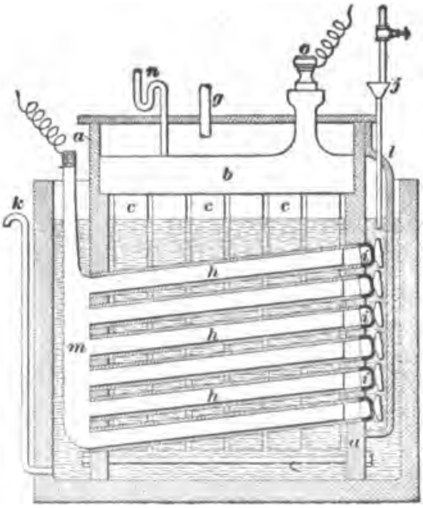


FIG. 23

83. Outhenin-Chalandre Cell.—This consists of an inner closed anode cell *a*, Fig. 23, containing round carbon anodes *c*, connected, by means of the lead support *b*, with the terminal *o*. A lead pipe *g* passing through the top carries the chlorine gas to the gas main. The sheet-iron cathodes *h* forming part of the plate *m*, the top of which serves as the terminal, are contained in sloping, porous tubes, which are hermetically sealed through the walls of the anode chamber, but which allow free circulation of cathode liquid through them. The tubes are fastened in place by an arrangement at *i*. The anodes are suspended in rows of six between adjacent sets of cathode tubes. Water is admitted to cathode chamber *j* and caustic-soda solution drawn off at *k*. The anode solution is sodium chloride, and the cathode solution sodium hydrate. The hydrogen produced at the cathode passes up the tubes and is collected in the hood *l*. It is then conducted away and used for other purposes.

The anode cell is filled through pipe *n*. It is claimed for this process that soda can be made fairly concentrated. It is also claimed that a sample of soda made by this process contained 97.5 per cent. caustic soda (dry basis).

The chief advantages of this cell are the complete separation of soda and chlorine and the collection and utilization of the hydrogen; but on account of the diaphragm, a high electromotive force is necessary between the terminals. In some mills an evaporator somewhat similar to the Yaryan is employed to remove the salt from the caustic liquor.

84. Treatment of Bleached Stock. — The bleached and washed stock, in case the washing is not done in the beaters, follows one of the following courses: It is (1) transferred to the mixers; (2) transferred to the wet machine; (3) transferred to the pulp machine; or, (4) transferred directly to the beating engine.

1. The object of the mixers (which are large, cylindrical tanks provided with agitators in the center, and capable of holding several bleachers of stock) is to furnish uniform stock for the beaters, in case the pulp is worked right up into paper. The stock is passing in and out of the mixers continually.

2. The wet machine, similar to the one described under the head of "Soda Process," is used in case it is desired to transfer or ship the pulp in folds, or to weigh the amount used in the beaters. This is usually customary in the

treatment of the sulphite fiber, after which it contains about 65 per cent. moisture. It is transferred to the beater in folds and opened out before charging same.

3. Before passing over the pulp machine, the pulp first passes through a screen similar to the one described under "Soda Process," from the floor box of which it passes up through a number of holes into a vat, where it is mixed with more water. There is a wire-covered roll revolving in this vat, by which the pulp is taken up and transferred to a felt, as in the wet machine, and conveyed over one or more suction boxes through a series of press rolls and over the driers (the suction boxes, driers, and press rolls being similar to those of a paper machine, to be described later). It is wound on a long reel at the end of the machine and finally slit and rewound in rolls of a convenient size to handle (100 to 150 pounds each). There are usually several reels so arranged that while cutting one another is winding, and thus the pulp is run continually. These rolls are

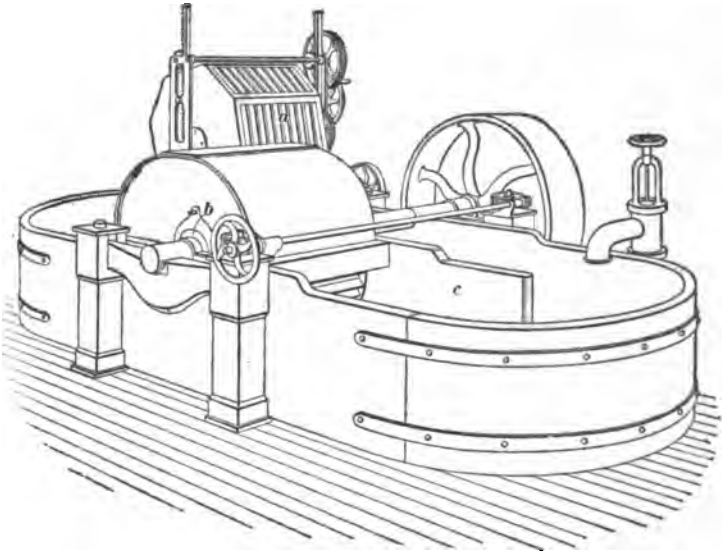


FIG. 24

weighed and tied up for shipment, and contain about 7 to 8 per cent. moisture. The rolls are either chopped up and put into the beaters in sheets or allowed to run in from a spindle.

4. The stock is pumped directly from the drainers to the beaters, where it is finally mixed for the paper machines.

BEATING

85. Beating Engine.—This machine, which has been referred to before, is shown in Fig. 24. It is made of wood

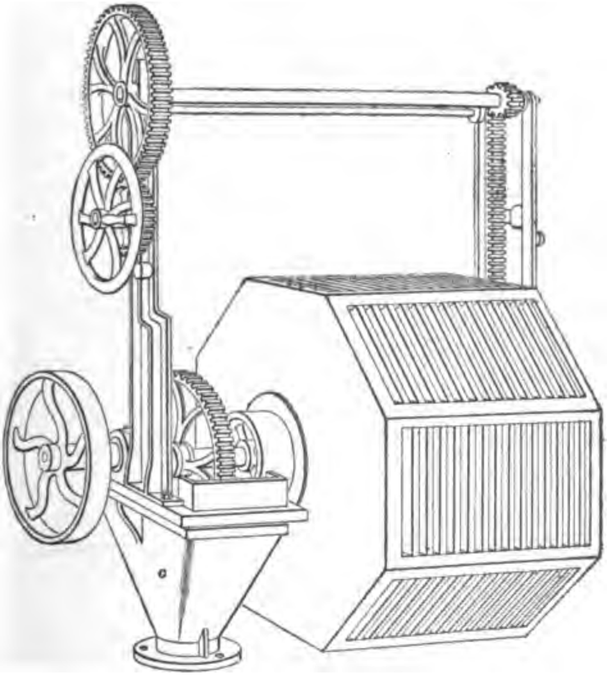


FIG. 25

or iron and provided with a washer *a*, a bedplate and roll *b*, and a midfeather *c*, as in the breaking engine previously described. The washer, which is shown in Figs. 25 and 26, consists of an octagonal drum, the faces of which are made of lattice work, admitting of the free passage of water to the interior, while the ends are closed in such a manner as to allow the water to pass out through the axis only. This drum is covered with a fine-mesh wire cloth, which prevents the carrying off of fiber with the wash water. The interior of the drum, shown in cross-section in Fig. 26, is so arranged that in revolving, the arms *a* take up the water and carry it to the axis of the drum, through which it is conveyed to the trough *c*, shown in Fig. 25.

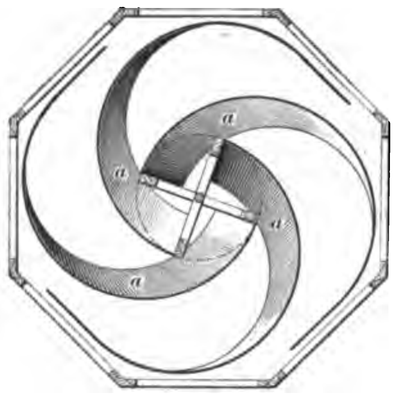


FIG. 26

86. The engine roll in Fig. 27 is provided with projecting steel knives, which are tapered in the opposite direction

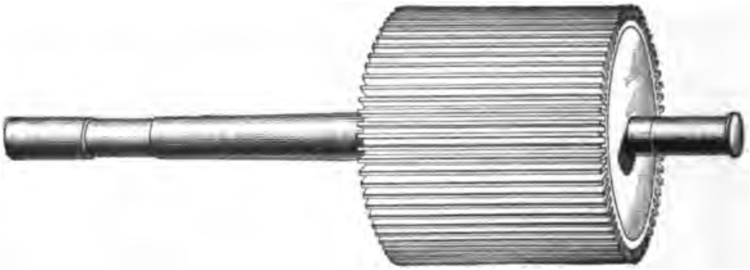


FIG. 27

from the knives in the bedplate, placed under the roll in the beater, so that when the roll revolves it produces a cutting action, similar to that of a pair of shears. There is a small depression in the floor of the beater, known as the *sand trap*, in which heavy particles of dirt, sand, etc., are caught during the beating operation. It is in this engine that the stock is prepared for the paper machine. By the beating and cutting action of the roll and bedplate, the fibers are separated and reduced in length, the fineness being regulated by varying the distance between the roll and bedplate. The fibers from straw do not require any beating ; those from esparto and wood require considerable, while the fibers from rags require excessive beating and cutting, on account of their length.

87. The beating is carried on by gradually lowering the roll, until the required effect is produced. In order to produce papers of the quality required, it is necessary to mix various fibers, which is usually done in the beating engine. In mixing these various fibers, such as rags with esparto, rags with wood, esparto with straw (for newspaper), sulphite stock with soda stock, ground wood with other fibers in preparing newspaper, etc., and, in fact, any of the fibers, the paper maker must use his best judgment, bearing in mind the different effects produced on the different fibers by the beating action, etc. If the various fibers were blended as above suggested, without the addition of any foreign materials, and run out into sheets, the product would be blotting paper.

88. Broke Beater.—This machine, as its name suggests, is used in working up *broke*, which is partially formed paper, obtained when starting the paper machine; paper damaged in passing over the drying cylinders; and imperfect or rejected paper. This engine is the same as the ordinary beating engine, excepting that a steam line enters it by which the stock is highly heated. It is usually customary to add a little caustic soda to the stock, to assist in breaking it up again.

89. Jordan Engine. — This is an improved form of engine, which, when used, will save from one-third to one-half the time required for beating, when done in the beating engine. The working of the Jordan engine can be plainly understood by reference to Figs. 28 and 29, which give a view of the Horne-Jordan engine set up and also the interior structure. It consists of a cast-iron cone *a*, which fits in the cone *b*, forming the body of the engine, and revolves at a speed of from 350 to 400 revolutions per minute. Both cones are fitted with angled steel knives, held in position by hardwood wedges. The plate *c* is bolted on tightly and the packing gland *d* adjusted over the shaft. The arrangement *e* is attached as shown in Fig. 28, and by means of the screw arrangement *f*, the cones can be adjusted so as to regulate the fineness of the stock. This form of Jordan is the one in most general use, although there are others that are giving very good results, among which the

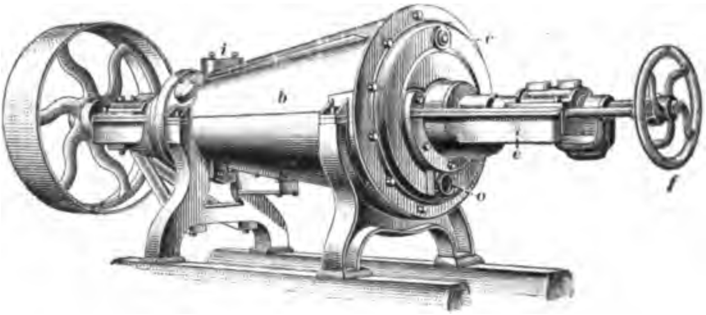


FIG. 28

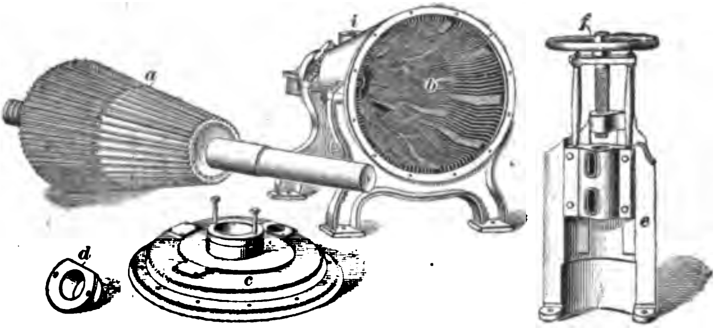


FIG. 29

Marshall perfecting engine might be mentioned, which differs from the Jordan in having rims on the end of each of the cones, set with knives, and accomplishes the cutting at this point also.

The half-beaten stock from the beating engine is pumped into a supply chest over the Jordan. From this chest it is furnished, by means of a pipe, the stock entering the engine at *i*, the finished stock leaving the engine at *o*, and is conveyed by means of a pipe to the stuff chest, from which it is pumped to the screens of the paper machines.

SIZING, LOADING, AND COLORING

SIZING

90. It is necessary that writing paper, book paper, etc., shall be more or less ink or waterproof, so that when used for writing or printing the ink will not spread, but leave good, plain characters. This property is imparted to the paper by the use of what are known as *sizing agents*, which are assisted to some extent by the *loading agents*, to be considered later.

91. Engine Sizing.—This is accomplished by precipitating *rosin size* with alum, or some other precipitant, upon the fiber in the beating engine.

92. Rosin Size.—This size is prepared in a number of different ways, but all accomplish the same purpose—that is, getting the rosin in such shape that it can be made into a solution, from which it is again precipitated as above suggested. There has been a great amount of discussion as to what is the true sizing agent, some of the authorities contending that the free rosin is the only sizing agent, others that the resinate of aluminum is the true sizing agent, while still others contend that it is due to both. We are from practical experience inclined to favor the view that the sizing is as much due to resinate of aluminum as to the free rosin, and for some time used sizing in which the amount of free rosin varied from 3 per cent. to almost 35 per cent. When using a size containing 3 per cent. of free rosin, a larger amount of alum was required to precipitate the size, but no more rosin was required to furnish as hard-sized papers than when 35 per cent. of free rosin was used. The chief advantages of using a size containing a large amount of free rosin are (1) the saving of considerable soda in making up the rosin soap, and (2) the saving of an enormous amount of alum or other precipitant used.

93. Brown Size.—This size is prepared by about the same method in the different mills, but different proportions are used. The rosin is first crushed, then melted in a separate kettle, and run in to a hot solution of soda in the mixing kettle. The boiling is continued cautiously until the rosin is saponified, the kettle being heated either by live steam or a steam coil. If the size shows a tendency to boil over, this can be kept down by sprinkling a little cold water on it. The size should be well stirred during the boiling operation. The amount of soda varies from 18 to 22 pounds per 100 pounds of rosin. Care should be taken not to use too much water, and the aim should be to keep the mixture at such a density that the size will float, while the dirt sinks to the bottom. The heavy size contains about 40 per cent. of water. The finished size should be drawn off and allowed to stand for about a week before using. When using, it is customary to dilute to a very thin solution containing over over 85 per cent. of water.

94. White Size.—A very good method for the preparation of white size is the one patented by H. Hampel and Victor Zampis, of Vienna. It has been introduced to some extent into this country. The apparatus for the preparation of this size consists of a large oblong tank, which has an agitator consisting of wings or paddles, revolving on a horizontal shaft. The cooking is not done under pressure. These tanks are of various sizes, but the proportions given below are for a tank holding 1 ton of rosin. The tank is

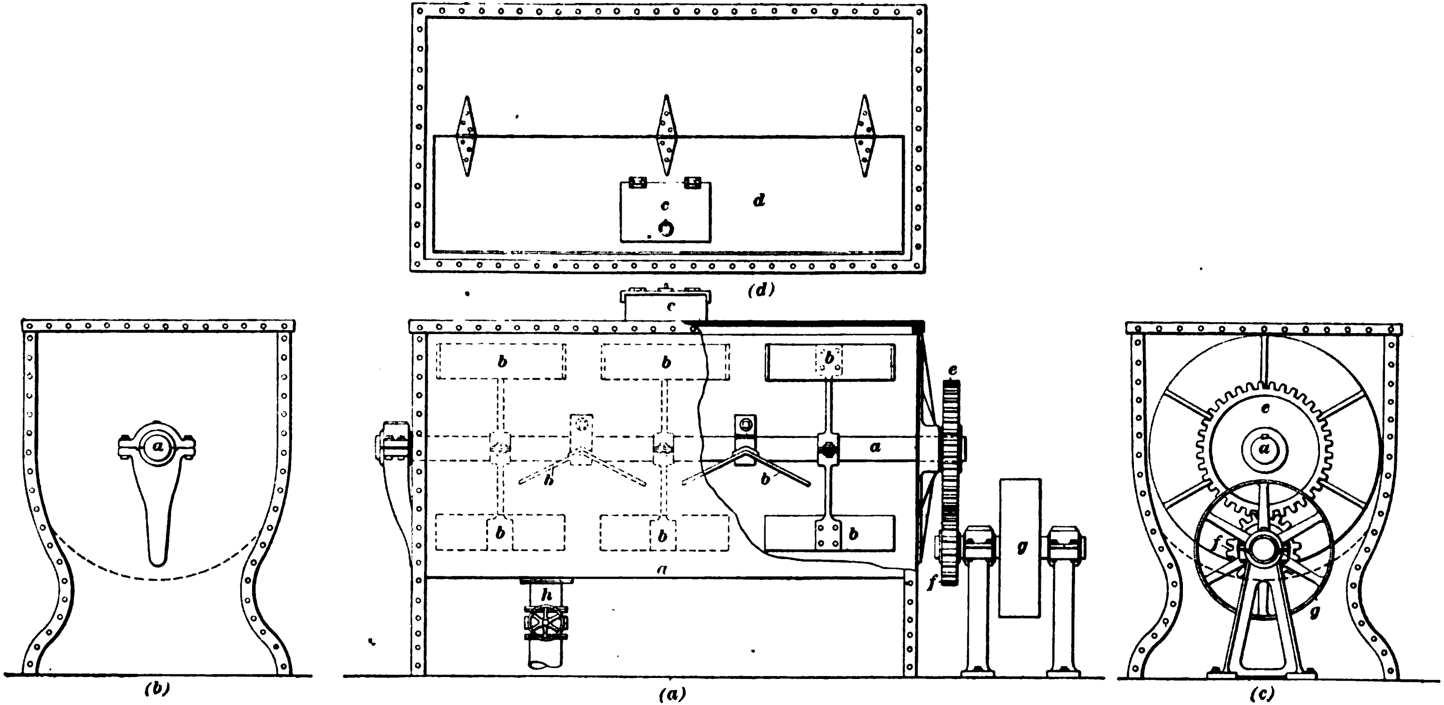

d
c
(d)
a
b
b
b
b
b
b
b
b
c
a
e
f
g
h
(a)
a
(b)
e
a
f
g
(c)

FIG. 30

covered by means of a large lid, which has a small manhole in the top, for introducing the ingredients, which can also be covered.

Fig. 30 gives a side view (*a*) and two ends views (*b*) and (*c*), also top view (*d*), of the tank used. In the figures, *a* is the shaft on which the agitator paddles *b* are attached. The shaft is run by the cog wheel *e*, which is in turn run by the smaller cog wheel *f*, which is attached to the pulley *g*. There is a small opening *c* in the large door *d* on the top of the tank, and it is through this small door that the charge is added. When the cook is ready to dump, the valve *h* is opened. The dimensions of the tank, as given in the figure, are 4 feet 3 inches × 2 feet × 2 feet.

95. In making up the heavy size, 31 gallons of water are brought to a boil, the agitator started, 60 pounds of soda ash added, and the agitation continued for some minutes, to dissolve the soda. The steam is then shut off and 400 pounds of rosin, which has previously been finely crushed, slowly added. The steam is then turned on gradually, and the balance of the 2,000 pounds of rosin added, taking care that there is enough steam on to keep the rosin from getting too thick. The temperature is brought up to 180° F., the steam shut off, and 160 pounds of soda ash cautiously added. After the soda ash is all in, it is agitated without steam for about 4 hours, and then brought almost to a boil, being kept at this temperature for about ½ hour, after which the steam is shut off, the agitator stopped, and the size dumped into the storage tank below. One cook will make about 300 gallons of heavy size.

The white size is made by mixing about 60 gallons of the heavy size with 2,000 gallons of water, which has been heated to about 175° F., but the steam is shut off before adding the size. This is agitated for about ½ hour and allowed to cool before using.

96. Another method of preparing white size is by boiling rosin with alkali and water, with continual stirring, under pressure, thus preventing the escape of the easily volatile resins. The carbon dioxide generated is also retained, which, it is claimed, improves the size. When the cooking is complete, the rosin size is driven to the storage tanks by means of the pressure remaining in the boiler. Before adding the size to the stock, the excessive amount of water is removed from the latter by means of the drum washers and the washer raised. The amount of size necessary is strained into the beaters in order to keep out any dirt that would otherwise enter the paper through this source. After adding the size, the stock should be allowed to circulate a couple of times, and the required amount of alum added, which precipitates the combined rosin as resinate of aluminum, with the production of sodium sulphate.

$$3Na_2R + Al_2(SO_4)_3 = 3Na_2SO_4 + Al_2R_3$$

In the above reaction R denotes resinic acids. The alum may be added in the dry state, but it is customary to first dissolve it, and knowing the strength of the solution, the amount required can be measured. The amount of alum necessary depends on the amount of size used and the proportion of free rosin in the same.

97. Animal Size or Glue.—This is mixed with water at a temperature of about 190° F. for from ten to fifteen hours, a little alum being generally added. This size is sometimes added to the stock in the beaters just before dumping, but generally added by a method called *tub sizing* on the machines, which will be described later.

98. Other Sizes.—Starch is used to some extent as an auxiliary sizing agent, as are also sodium aluminate, sodium silicate, and casein, which is made from milk. The last mentioned, casein is largely applied on the machine, and is used to give a coating or better finish to the paper. It is also used in about a 40-per-cent. solution in the beating engines.

99. Adaptation of the Various Sizes.—The principal adaptations of the different sizing agents are as follows:

Rosin size is used to fill up the pores in the paper between the fibers and to make it waterproof, thus keeping ink from running to a greater or lesser extent, depending on the amount used.

Animal size is used mostly as a surface size, and makes the paper still better in the property of preventing ink from spreading when used in connection with rosin size. It gives the paper a better surface.

Casein size is of great value as a paper coating, in which capacity it is mostly used, giving the paper a nice finish.

Starch at the present time is used mostly as a filler, making the paper stronger. It is claimed that the paper in which starch is used has a better surface and feel. The value of the use of starch is very doubtful, as it is used in such small quantities.

Sodium aluminate is used sometimes in place of sodium carbonate in preparing size. The size is added to the pulp in the usual manner and precipitated with either magnesium chloride or sulphate. Rosin, magnesia, and alumina are all precipitated at the same time, which is claimed to be an advantage.

Sodium silicate is used when a hard paper is desired, which has a rattle. It is strongly caustic and can be used in place of sodium carbonate or may be mixed with the size in the engine. When alum is added, a bulky, gelatinous precipitate of hydrated silicic acid is formed, similar to precipitated alumina. Its use will produce a good, hard writing paper.

LOADING

100. In the manufacture of almost any kind of paper, excepting the very highest quality, it is customary to add some cheap material, such as china clay, agalite, pearl hardening (sulphate of calcium), etc., to the stock in the beaters, which will give it weight and also make it more compact. In some mills it is customary to mix these materials in water, which is kept agitated continually, the strength of which mixture is known and the amount used measured. However, quite a number of mills add the filler in the dry state. When using clay in the dry state, it is usually in lumps. It is better to add this material when furnishing the beater, as it will then have more time to work up into the stock. When used in the mixed form, the loading materials are in some mills added after the sizing agents, while in others before them. Agalite, on account of its fibrous nature, is retained much better than clay.

COLORING

101. Coloring is effected by addition of dyestuffs or pigments to the stock in the beaters after sizing and loading. There are a great variety of coloring matters, which are mixed with the stock to produce various shades in the finished paper. A yellow tint in the stock is neutralized by addition of red and blue. The blues generally used are ultramarine, smalts, and various aniline blues, while the reds are usually prepared from cochineal or aniline dyes, but as the latter are affected by the use of alum, the former is to be preferred. Alizarine and red ocher (oxide of iron) are also used to a considerable extent in giving red tints.

Yellows are produced by the use of yellow ocher (oxide of iron), chromate of lead, and some of the coal-tar dyes, the principal ones being naphthol yellow and auramine. Browns are produced by the use of a number of pigments of the iron oxides, or the Bismarck browns (salts of triamido azobenzene). Greens are usually produced by means of malachite green or Victoria green.

It is better to mix the coloring matters with water before adding them to the stock in the beater. The aniline blues should be dissolved in hot water and then diluted. Carmine should be dissolved in a little ammonia water and then diluted. Samples of the pulp treated are taken from time to time and matched against a sample of the paper that the paper maker is running, and the practiced eye of the beaterman can readily determine when the desired effect is produced.

Before attempting to size or color the stock, it should be perfectly cold, otherwise good results cannot be obtained. When the stock has been uniformly colored and well worked up in the beaters, it is passed to the Jordan engine, where it is finally prepared for the paper machines.

THE MANUFACTURE OF PAPER FROM PULP

102. General Remarks.—The pulp made by the various processes already described is now in suitable condition for making into paper. For some fine grades of paper, this is done by hand, but by far the greatest amount of paper used is machine made. Both processes will be described.

PROCESS FOR MAKING PAPER BY HAND

103. In making paper by hand, the stock passes from the beaters to vats, which are 5 feet square and 4 feet deep, provided with a steam pipe, to keep the pulp at the required temperature for working, and with an agitator to keep the pulp and water well mixed. The stock is taken from the vat by hand in a mold, consisting of a frame, covered first with heavy wire and then with a fine-mesh wire, upon which the sheet is formed. A movable frame is fitted upon the outside of the mold, which extends a little above the wire, forming a sort of wire-bottom tray. The movable frame, termed the deckle, forms the edges of the paper, and should fit snugly to the frame. The stock is taken up in the mold, which is then shaken, the deckle removed, and the mold passed to another workman, who turns the sheet on a piece of felt, while the vat man forms another sheet. Felt is laid between all the sheets, and when about 50 deep, they are removed to a press, where the water is squeezed out. The sheets are then removed from the felts and either hung up or laid out on a board to dry. They are then sized by spreading out the sheets in a vat of animal size, after which they are dried slowly, pressed again (better by running through calender rolls), and passed to the finishing room, where specks are picked out, the imperfect sheets separated, and the balance counted and packed for shipment. Water-marked paper can be made by having the desired mark worked in wire on the bottom of the mold.

PROCESS OF MAKING PAPER BY MACHINE

104. Paper Machine, or Fourdrinier.—This machine consists of a fine-mesh endless-wire cloth, upon which the stock forms into a sheet, the width of which is regulated by means of a rubber strap called a *deckle strap*, shown at *a*, Fig. 31. The water passes through the wire, and the formed sheet is carried by means of a felt through the press rolls and thence over the driers and heated calenders to the reels.

The stock coming from the Jordans or from the beaters passes into a large cylindrical tank, called the *stuff chest*, which is made of wood or iron and provided with an agitator, extending to the bottom of the tank, which is kept moving at a moderate speed. The stock is pumped from the stuff chest to the regulating box, a small box which is constantly kept filled by means of the inlet pipe at the bottom and an overflow pipe near the top, which carries the excess of stock back to the stuff chest. The discharge pipe to the screens is near the bottom of the regulating box, so that there is a uniform pressure at all times, and the amount of stock furnished can be regulated by means of a cock on this pipe. In some mills, the stock passes from the regulating box over what is termed a *sand table*, which is a long, shallow box, with felt-covered bottom, having strips of wood placed across the direction of flow of the pulp. The object of this table is to hold back sand, etc. that has escaped removal by previous treatment. The stock then passes to the screens. However, it is not always customary to make use of the sand table, the stock going directly from the regulating box to the screens.

105. The screens are similar to those described under the soda process and are to remove any foreign matter, etc

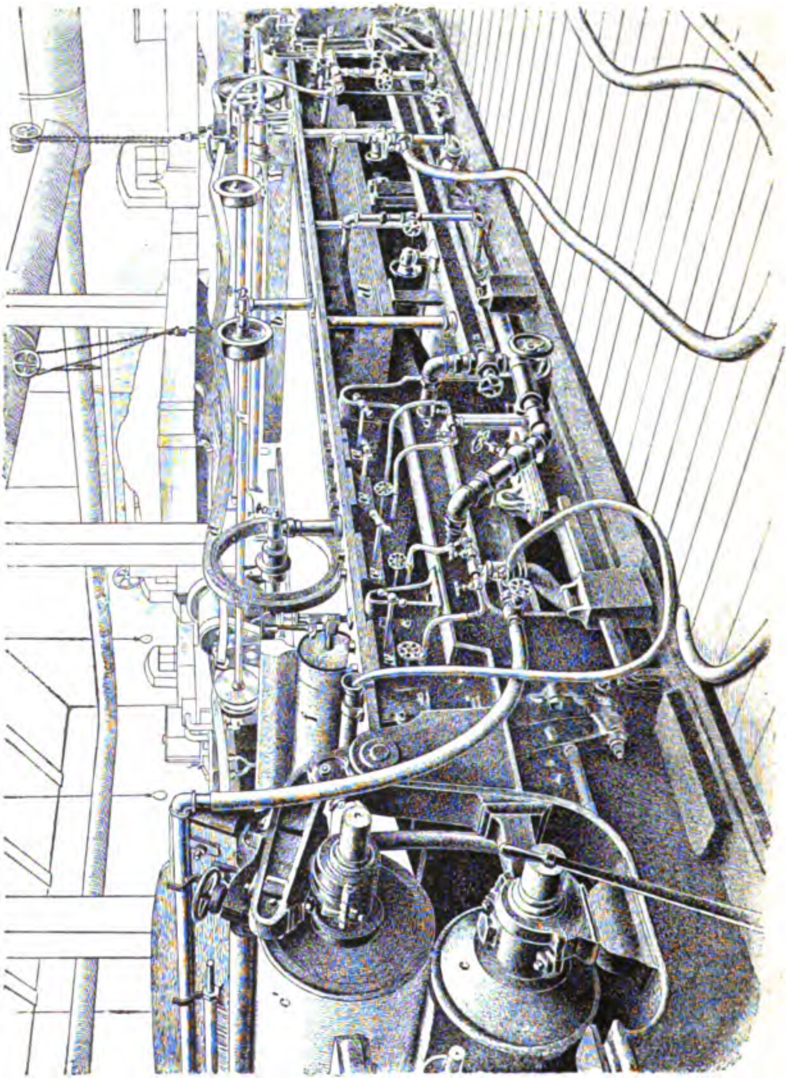


FIG. 31

that has escaped removal up to this point. After passing through the screens the stock should be free from knots and dirt and ready for making into paper. After leaving the screens, the stock usually flows down into what is called the **head box,** entering the bottom, where it is mixed with more water, which enters from a supply pipe. This box serves to mix the stock well before it flows on the wire, which it does by overflowing the box. It also serves to catch any heavy particles of matter, as they will remain at the bottom and can be washed out from time to time.

106. The uniform thickness of the sheet is regulated by a gate called the slicer, which is made of two pieces of brass, bolted together in the middle, so that it can be lengthened or shortened, according to the width of the sheet. This slicer is placed right where the stock is passed on the wire, and can be regulated at different heights from the wire, according to the thickness of the sheet required, by means of screws, which height must be the same all the way across, in order to make a sheet of uniform thickness. The slicer is shown at *b* in Fig. 31.

107. The endless wire used is from 60 to 70 mesh, and is joined by being carefully sewed together with wire. The length of the wire is from 35 to 40 feet, and the width is usually from 100 to 130 inches, though they are made as wide as 160 inches. The wire passes around the lower couch roll *c*, Fig. 31, down under the *save-all*, and back to the breast roll. In its passage over the surface, the wire is supported by a large number of small brass rolls, and in returning to the breast roll it passes over several small ones. The frame has an attachment by which the wire is given a shaking motion from side to side, which serves to weave the fibers in their passage over the wire. Under the wire is situated the save-all, which is a shallow box *d*, Fig. 31, into which the waste water coming through the wire drops, and is again used in place of fresh water for diluting the stock in the head box referred to above.

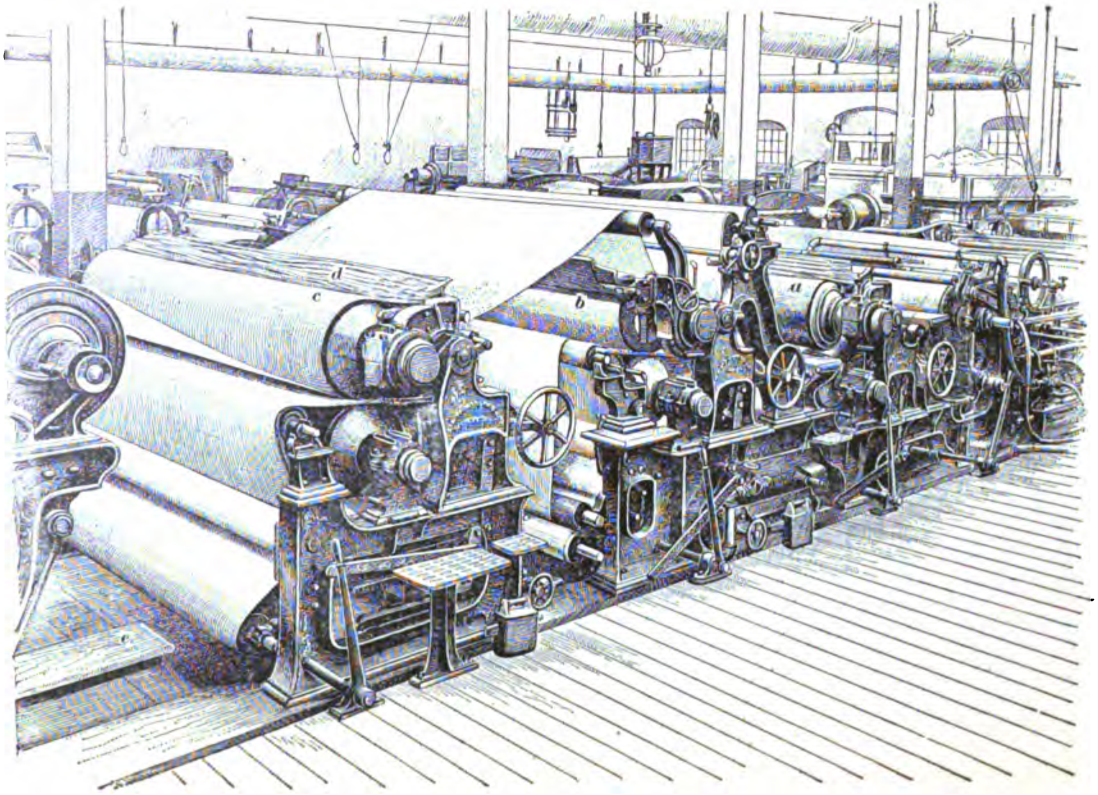


FIG. 28

108. Near the end of the wire and under it are situated the suction boxes *e*, four of which are shown in Fig. 31. These are long, narrow boxes, which extend across the whole width of the wire, and are connected with the vacuum pump. The surface of the box, which is very smooth, is perforated, and the water is further removed from the stock by having the wire pass over it. There is a screw arrangement at the end of the box, by which plugs are moved forwards or backwards according to the width of the paper, so that the boxes will not lose their suction. These plugs are always kept in as far as the deckle straps.

The deckle straps, *a*, Fig. 31, are heavy, square, rubber bands, which rest on the wire and are carried along with it, thus regulating the width of the sheet.

109. The *dandy roll f*, Fig. 31, is situated near the end of the wire, and is used for making the "water mark" in the paper. It consists of a skeleton roll covered with a wire cloth upon which the desired design is worked with fine wire. If the paper is required to be alike on both sides, with no special design, the roll is only covered with wire cloth, the impression of which corresponds with the impression on the wire cloth. This paper is known as *wove paper*. *Laid paper* is made by the dandy having a number of equidistant transverse wires upon its upper surface.

110. The paper passes under the dandy roll and is carried by the wire cloth between the couch rolls *c*, *c'*, Fig. 31, which are brass or wooden cylinders with a jacket of felt. On some machines the top roll is wood and the bottom roll is iron covered with a heavy layer of rubber. There is a screw arrangement above the top roll, by means of which the pressure can be regulated, thereby pressing the water out of the paper in its passage between the rolls. The paper is then carried, by means of felts and assistance of the experienced machine tender, through the press rolls proper. The number of press rolls varies on different machines, the machine shown in Fig. 32 having three sets, *a*, *b*, and *c*.

The top roll of each set is provided with what is termed a *doctor d*, which keeps the roll clean, scraping off the pulp that sticks to it. The pressure on these rolls is also regulated by a screw arrangement. After passing through the last press roll, the sheet is transferred by the machine tender across an open space to the driers, or drying cylinders; this space *c* is wide enough for the machine tender to pass back and forth under the sheet.

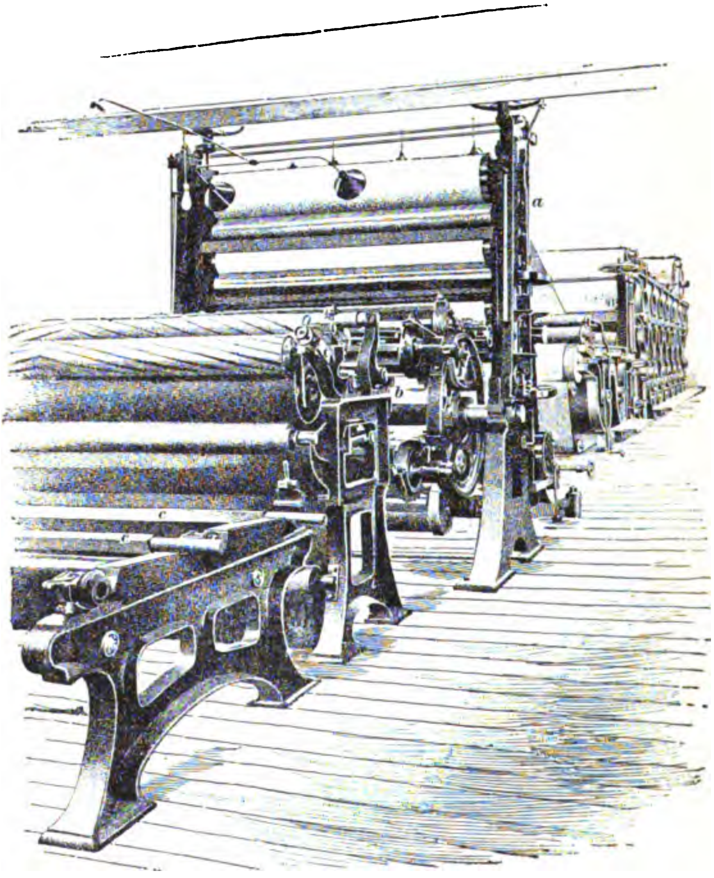


FIG. 33

111. These drying cylinders are large, hollow rolls, which are heated by means of steam. The paper is carried by means of a drier felt over the series of drying cylinders, which vary in number on different machines, and can be seen in the distance, Fig. 33.

112. Tub Sizing.—After the paper has passed over a couple of the drying cylinders, it passes through a vat of liquid animal size and then between two rollers, which squeeze out the excess of size. It is then wound on a reel. In some mills it is allowed to stand for a time and then passed from the reels over a series of wooden drums furnished with fans, by means of which the paper is dried slowly. The most general custom is to pass the paper from the squeeze rolls over another series of driers, and thus keep the sheet in continual motion.

After passing over the driers, the sheet is transferred to the calenders.

113. Calendering. — By this operation the paper is given a higher finish by pressing between rolls on a machine known as a *calender*. Calenders consist of a series of highly polished iron cylinders, having a screw arrangement at the top for regulating the pressure, the rolls being heated by steam. They vary in number on different machines, some having as high as three sets; the machine shown in Fig. 33 has but one set *a*. The paper passes over these hot rolls, acquiring a high finish, and thence to the reels, shown at *b*. There are usually six reels in a set, and after one has been wound to the desired thickness, the wheel is turned and the paper cut off and started on another reel. When rewinding into rolls, only one sheet is wound at a time, which is passed from the reel around a smooth, brass roll, through the slitters, which are small, sharp-edged, revolving wheels. The slitters are situated at equal intervals and divide the sheet into two or more sheets of the same size, which are rewound on cores, placed on shafts *c*, which are clamped down and made to revolve by means of a friction clutch at the other end.

When laying the paper in sheets at the end of the machine, as many as five reels are slit and cut at one time, as will be explained under the heading "Cutting the Paper."

By the use of the above paper machine, there is a continual sheet of paper forming, passing over the machine and winding.

114. Supercalendering. — It is sometimes necessary to have an extra-high finish on the paper, which is done on the glazing calenders, generally called *supercalenders*. These machines are also of various types, one being a series of rolls that are alternately of highly polished iron and compressed paper, the iron rolls being hollow and heated by steam. Another type consists of a stack of highly polished, chilled-iron rolls. The rolls are bored out and can be heated by steam, and there is an arrangement by which one or more of the rolls can be raised, according to the finish required.

The calender in most common use is shown in Fig. 34, and consists of a stack of rolls, which are alternately of highly polished iron and compressed paper, neither of which is heated by steam. These rolls are pressed down against each other by turning a wheel *a*, which operates the large wheels *b* at the top of the calenders. The lower roll is made to revolve at a high speed, and by means of the friction exerted on the second roll it revolves in the opposite direction, and in like manner all the other rolls revolve at a high speed. Owing to the great friction on these rolls, they become very hot. The iron and paper rolls are designated by the initials *i* and *p*, respectively, on their ends.

The roll of paper to be calendered is transferred from a small truck to the bar *d*, when by turning a crank at *c* the position of the roll is changed to *e*. The sheet is then passed up to a machine tender, who passes it under a brass roll *f*, and thence around the rolls of the calender, finally winding it again on a core placed on the rod at *d*. It is usually customary to have a perforated steam pipe extending across each side of the machine, so that a little steam is allowed to strike both sides of the sheet as it starts over the calenders.

The paper coming from the calenders has a very high finish, and is transferred to the slitting and rewinding machine.

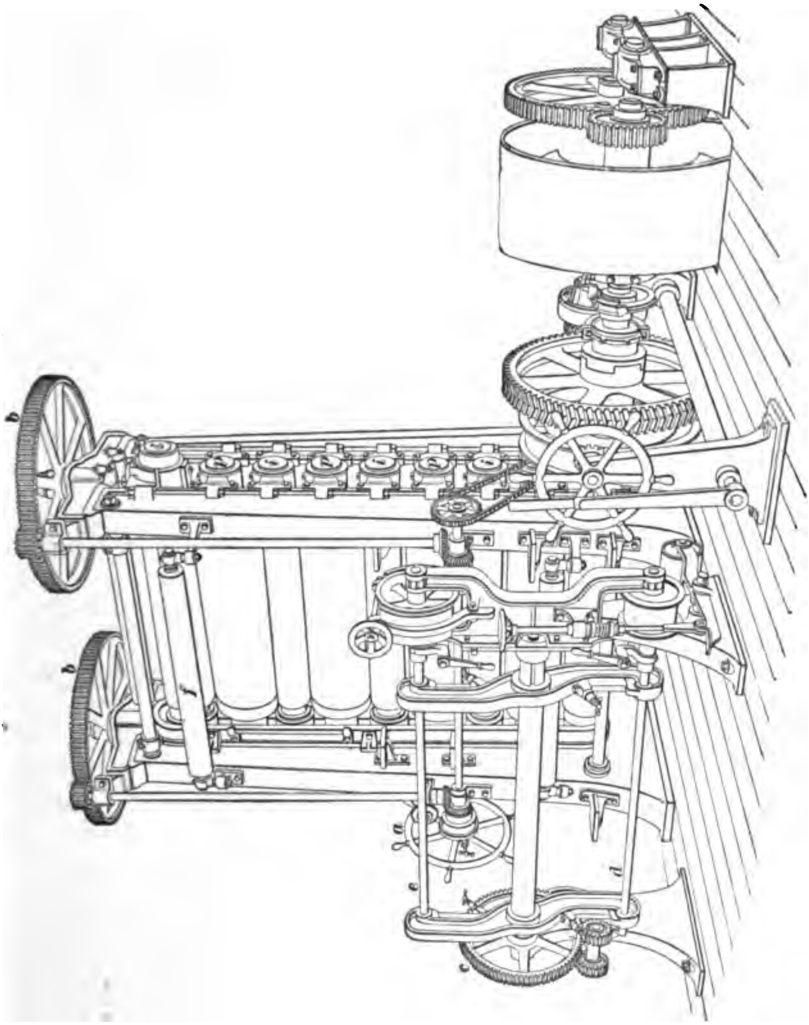
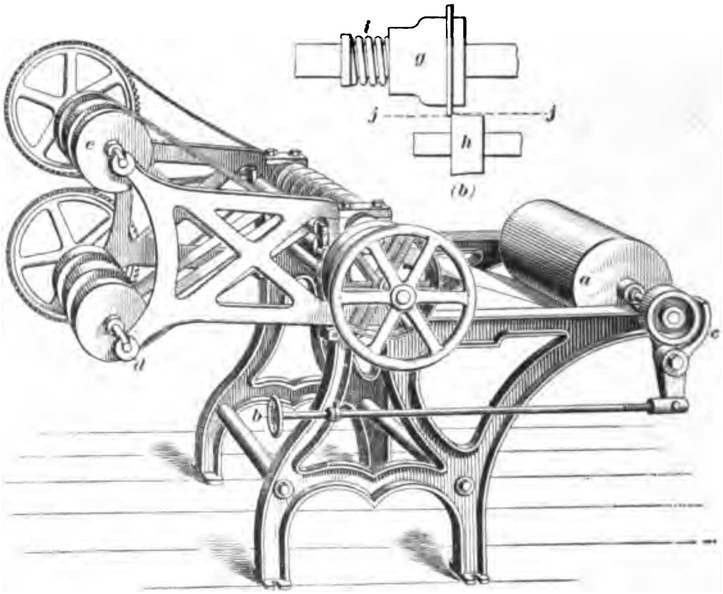


FIG. 34

115. Cutting the Paper.—This is very frequently done, as previously stated, at the end of the paper machine, where the large reels of paper are first slit by means of small, sharp-edged wheels, under which it passes before coming to the main knife, which is a knife projecting from a revolving drum, and extending across the whole width of the machine. It operates by passing closely against a dead knife, over which the paper passes. Upon every revolution of the drum, the paper which has been previously slit is cut into sheets of uniform size. These sheets drop upon strips of traveling felt, from which they are lifted by girls or boys and placed in stacks, which operation is termed *laying the paper*. These stacks are removed to trucks and conveyed to the finishing room. It is usually customary to slit and cut several different reels at the same time, and in place of one sheet, there are two or more sheets laid at one time in the same stack.



(a)

FIG. 35

When rewinding at the end of the machine, large rolls from the machine are generally slit into smaller rolls. When making paper that is to go to the supercalendering room, it must come from the paper machines in rolls, which, after supercalendering, are transferred to the cutting department, where they are cut into sheets and laid as above mentioned.

116. Slitting and Rewinding Machine.—In Fig. 35 (*a*) and (*b*) is shown a slitting and rewinding machine which slits and rewinds one large roll into six small ones.

The large roll is placed on a reel at *a*, which reel can be made to run easy or hard as desired by turning the screw arrangement at *b*, which regulates the brake *c*. The end of the paper is then passed through the slitting knives, so arranged as to regulate the size of the smaller rolls desired, and thence to cores attached to the reels *d* and *e*, which are run by cogs and chains attached to cogs on the slitting-knife shaft. When the machine is in motion, the winding of the smaller rolls causes a continued pull on the sheet, which unwinds the large roll, so that by tightening the brake *c* the smaller rolls are rolled tighter.

The slitting knives are similar to those at the end of a paper machine, one pair of which is shown in Fig. 35 (*b*). The cutting surfaces are slightly hollowed out, so as to make them sharp. The knife *g* is held against *h* by the spring *i*. The sheet of paper passing through the knives is represented by *j j*.

117. The guillotine cutter is used for trimming the edges of paper and also for cutting a large pack of paper into smaller sheets. This is provided with a gauge by which the size of the sheet cut can be regulated. Fig. 36 shows a guillotine cutter.

The sheets of paper to be cut are placed upon the table *a* in packs from 3 to 4 inches thick, touching the back support *b*, which can be moved backwards or forwards by turning the wheel *d*. This support is connected by means of an upright rod *f* with an endless measuring tape *g*, and the size of the sheet is read off at the pointer in front of the tape. By giving the lever *e* a slight turn and pushing it to one side, it forces a friction clutch *i* against the pulley and sets the machine in motion. The clamp *c* and knife *h* descend, and when the clamp reaches the paper its progress is arrested, holding the paper tight while the knife passes on down through it. The machine is so regulated that as soon as the knife has passed through the paper, the clamp and knife return to their original position. This upward-and-downward motion is kept up as long as the friction is kept on; but as soon as the lever is allowed to resume its original position the machine stops.

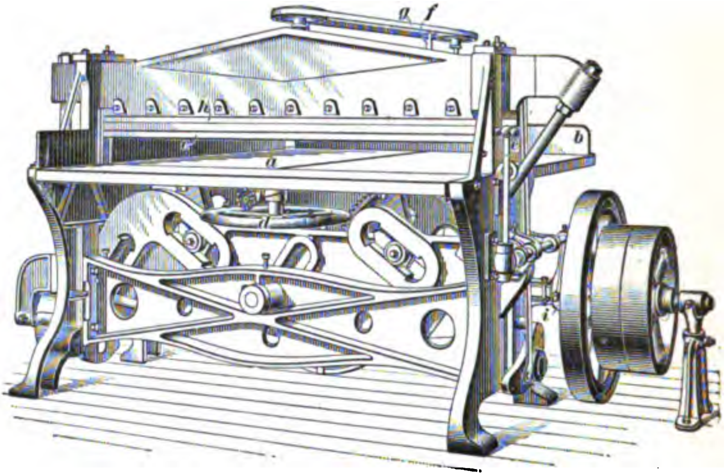


FIG. 86

118. Finishing the Paper.—After cutting, the paper is passed to the finishing room, where it is counted, sorted, and packed for shipment. The sheets are gone over by girls in this room and the defective ones thrown to one side and used up as "broke" or sold as an inferior quality of paper. The sheets that pass inspection are either packed in boxes or tied up with heavy wrapping paper, having wooden frames laid on each side so as to avoid being damaged in shipment. They are packed up in reams, which consist of from 480 to 516 sheets. The rolls are also well done up before shipping.

Papers are generally purchased by weight, and, therefore, the weight per ream is also expressed when distinguishing between them. Thus 26 × 40 – 80 – 500 means a ream of 500 sheets 26 inches × 40 inches, weighing 80 pounds.

PASTEBOARD OR CARDBOARD

119. General Remarks.—In the manufacture of boards, all kinds of refuse material occurring in the mill might be used, being sorted according to the quality of the board desired. After being well beaten it is mixed with a suitable amount of rag pulp, clay, etc. The boards are manufactured by superposing several sheets of paper and causing them to unite (1) by a sizing mixture; (2) by superposing several wet leaves at the time of couching; (3) by molds provided with thick deckles; or (4) by machines similar to paper machines, but having no drying cylinders, allowing them to dry in the open air.

A method for making cardboard with two different faces is as follows: The two pulps are mixed separately, and the first is run on the wire of a paper machine. When the water drains off to some extent, the second material is run on, highly diluted with water — better after the first has passed over the first suction box— the water from the second draining off through the first. The paper is then passed over the machine in the ordinary way.

A good cardboard is made from shoe linings, mixed with No. 1 linen rags, the larger amount being shoe linings. This is hard sized and has a smooth, erasable surface.

High-grade mill bristols are made of small percentage of rag pulp, the balance of wood pulp, mostly spruce. They are surface sized and allowed to dry.

Card middles, used for shoe boxes, after they are covered on one side with coated paper are made of spruce and ground wood on a cylinder machine.

Tag boards are made mostly from spruce sulphite, some of which have ground wood mixed in. Jute is also used to some extent.

Straw is used extensively in the manufacture of the cheaper quality of boards.

PARCHMENT PAPER

120. Parchment paper is made by dipping white unsized paper for half a minute in strong sulphuric acid of 60° Baumé and afterwards in water containing a little ammonia. The acid converts part of the cellulose into hydrocellulose, which gives the paper a gelatinous surface. Paper treated in this way becomes translucent and much stronger. In other methods, ammoniacal-cuprous-oxide solution or zinc chloride is used instead of sulphuric acid.

WATER AND ITS PURIFICATION

121. General Remarks.—Water is one of the most important factors in the manufacture of paper, as it is impossible to make good papers with the use of impure or dirty water. There is such a large quantity of water used around a paper mill that it furnishes quite a problem to purify it so that it will give the best results.

Almost every paper mill has in connection with it a filtering plant, the size of which varies with the amount of water used and the condition before filtering. If the water is very hard, it should undergo the usual treatment for boiler purposes; but for the manufacture of paper, the principal factor to be considered is the organic impurity. This causes the growth of what is termed *Algæ* in the water, which forms a kind of slime, coating the pipes and eventually causing the appearance of slime spots in the paper.

122. Filtration.—The aim of the paper maker is to remove the organic impurity from the water, which is very successfully accomplished by the use of the Warren or Jewel types of gravity filters. In both of these systems the water undergoes a preliminary treatment, as follows:

123. Alum Treatment.—The water is pumped up into a deep box, and before passing on to the settling basin it is treated with a solution of sulphate of aluminum, as follows: The alum is mixed up strong in one tank and then run into another tank, where it is diluted to the required strength. It then passes through the pipe *i*, Fig. 37, and valve *h* to a small oblong box *C*, from which box it is conveyed by the alum pump to the incoming water. The valve is provided with a ball float *g*, so arranged that it will close the valve and stop the flow of the solution of alum when the box is full, thus preventing the alum from overflowing. The faster the pump works, the more alum solution flows into the box, so that it is kept at the same level all the time.

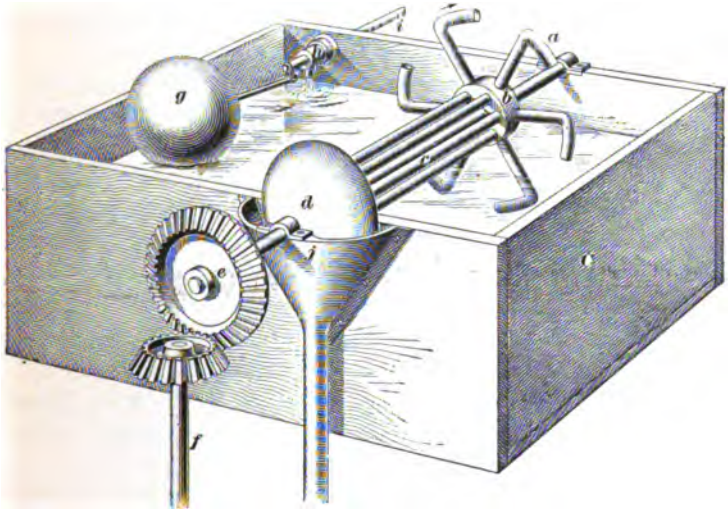


FIG. 37

The alum pump is operated by means of a turbine wheel, which is fastened to the side of the deep box mentioned above, right over the opening from the box to the narrow runway, so that all of the water entering the runway must pass through the turbine wheel. There is a gearing on the rod *f* at the turbine, which causes the rod to rotate, the speed varying with the speed of the turbine, and hence with the flow of water. The rod *f* has a similar gearing where it meets the gear-wheel *e* of the alum pump, which it causes to rotate. The pump consists of long, hollow arms *a* bent in the direction of the rotation. These arms when rotating pass through the alum solution, each one taking up its allotted portion of the same and conveying it to the drum *b*, from which it is conveyed by six long tubes *c* to the bell *d*, where it is discharged. The bell is so arranged that it is outside of the box *C*, and deflects the alum solution so that it is deposited in the funnel *j* at the side of the box and conveyed to the incoming water.

124. After treatment with the solution of alum, the water, when using the Warren system, passes through a long settling basin so arranged that it moves slowly out to the end of the basin, passes around a partition that extends down to the center of the basin, and returns on the other side. The size of this settling basin varies with the condition of the water to be treated. In some cases it requires from 45 minutes to 1 hour to make the passage around the basin, but it usually requires from 25 to 35 minutes.

The object of the settling basin is to give the alum plenty of time to act and thoroughly coagulate the organic matter, some of which settles to the bottom of the basin, while the remainder passes into the filters.

This is a very convenient arrangement, in that it looks after itself and supplies a uniform proportion of alum to the water, for as the flow of the water varies, the speed of the pump varies, and, therefore, the flow of the alum solution will vary. When the quantity of alum is to be reduced, it can be accomplished by plugging up one or more of the arms, hence the operator generally speaks of using so many "plugs" of alum, meaning the number of arms that have been left open. If the water contains considerable organic matter, it is customary to use about 1 grain of alum to every gallon of water filtered.

The following analysis shows the composition of a good alum for filter-plant use:

Sulphate of aluminum	53.98%
Sulphate of iron	.42%
Insoluble	.71%
Free alumina	.52%
Water	44.37%
Total	100.00%

There are several forms of pressure filters used in some mills, but the gravity filters are the ones most generally adopted.

125. Warren Filter.—This filter, shown in Fig. 38, is usually constructed of wood, and is 8 feet in diameter, having a bed of 50 square feet area, which contains from 20 to 24 inches of quartz sand *c*, supported on a perforated copper bottom *b*. The unfiltered water from the settling basin enters from the main *j* through the valve *e*, passes up into the filter tank *a*, and thence downwards through the bed of sand *c*, the perforated plate *b*, and through *f* to the filtered water main *i*, through which it is conducted to the storage tank. When it becomes necessary to clean the filter, which will be evident from the sluggish action of same, the valve *e* is closed, shutting off the unfiltered water, and the valve on pipe *g* opened, allowing the water in tank *a* to pass into the sewer. When the level in *a* falls, the water flows back from main *i* up through the bed of sand *c*, passing down the space *p* and gutter *n*, thence through waste pipe *g*. While this operation is going on, the agitator *d*, which was set in motion as soon as the water commenced to flow back through *i*, revolves by means of the mechanical arrangement *k*. While the agitator is in motion, the teeth of the rake stirs up all the sand, being lowered mechanically by the screw *m*. When the water flowing up through the bed becomes clear, the agitator is raised, the waste pipe closed, and when the tank is partially filled, the valve *e* is opened and filtration carried on as before.

Each 8-foot filter, when using alum, has a capacity of about 250,000 gallons in 24 hours.

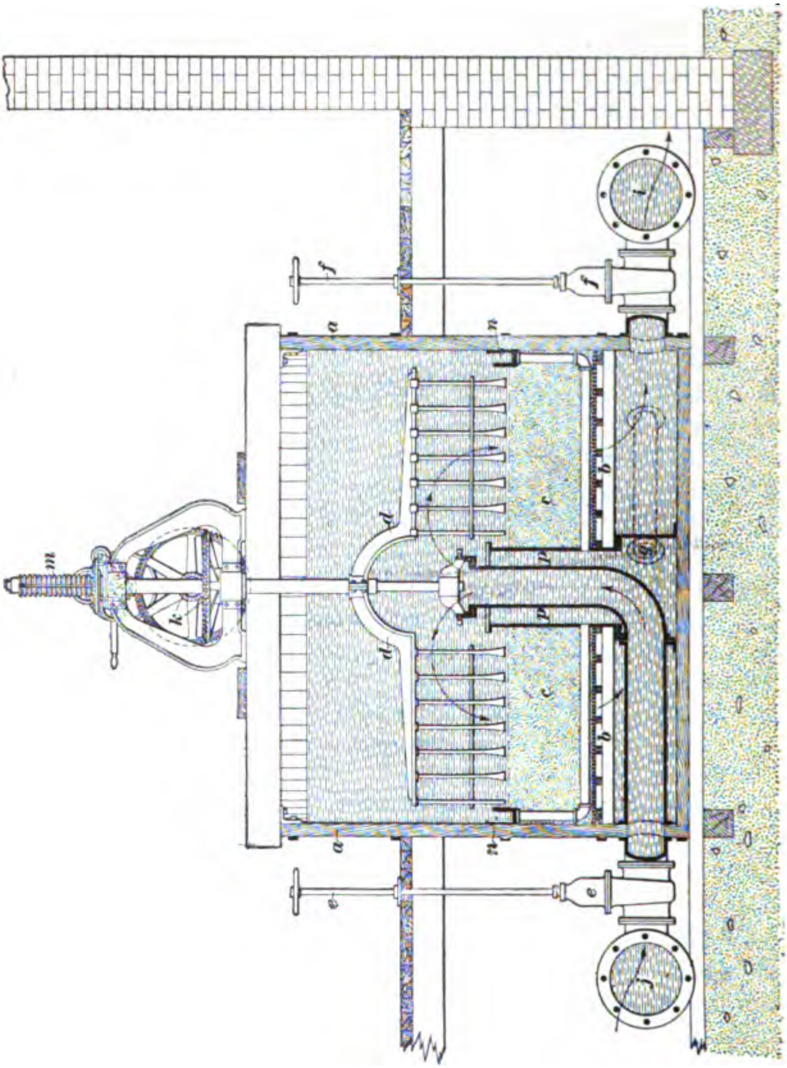

m
k
f
f
a
a
n
n
d
d
c
c
b
b
p
p
g
i
e
e
j

FIG. 38

126. Jewell Filter.—In this type of filter, shown in Fig. 39, the water coming direct from alum treatment enters the subsidence basin *a* through inlet valve *i*, which is located a short distance above the bottom. It is deflected in front of the inlet valve and flows slowly around the basin, and thus deposits a great deal of the sediment before passing through the filter bed, which it does by passing up through the central stand pipe *b* and overflowing it, there being enough water kept above the sand to keep from cutting channels through it. It differs from the Warren filter in being a double tank, and in place of having a perforated plate, it has a bottom made up of a series of small strainers, which empty into small horizontal pipes. These pipes empty into larger ones *c*, and thence into the main outlet pipe *h*. These strainers catch what sediment passes through the sand.

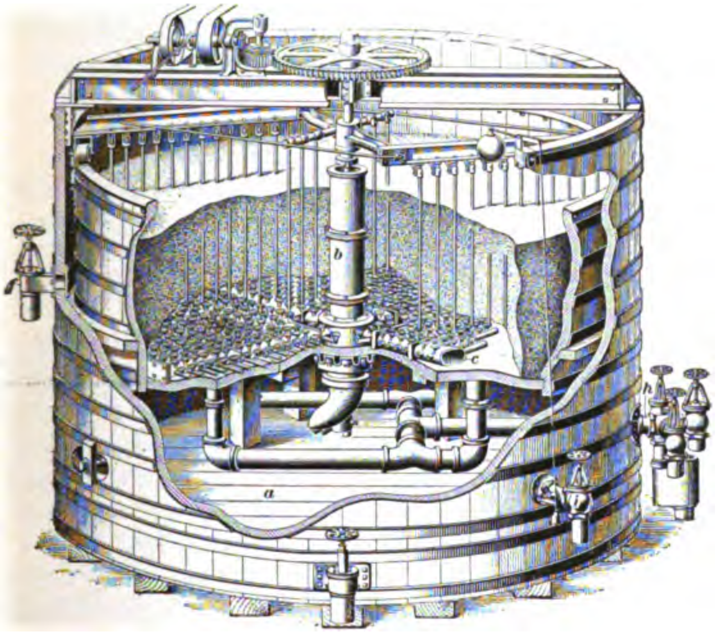


FIG. 39

In washing, the agitator is started, the rakes of which stir up the sand, and the water passing through it flows over the side of the inner tank into the outer tank, and thence out at the valve *f*.

127. The coagulant used in both systems is generally sulphate of aluminum, which is precipitated as hydrate by the alkalinity of the water, which, if not alkaline enough, should have a small amount of caustic lime added to it. This flocculent hydrate coagulates the organic matter present in the water and carries it down, thus freeing the water of its impurity.

MANUFACTURE OF PAPER

(PART 2)

ANALYSES AND TESTS OF MATERIALS USED AND OF FINISHED PRODUCTS

APPARATUS AND CHEMICALS

1. General Remarks.—It will be the object under the above heading to set forth methods for the analysis of the chemicals, liquors, etc. used, and at the same time give several quick tests, by the assistance of which the manufacturer is enabled to operate his plant on a more economical basis.

It is important that the chemist in charge of this department should have on hand from the outset the necessary apparatus and chemicals that he is likely to be called on to use at any time. In order to give an idea as to what will be required, we give herewith a list of apparatus and chemicals that will be necessary.

2. Apparatus.—The following apparatus will be found necessary for the analytical work herein described: A sensitive balance (enclosed in a glass case, the interior of which should be well desiccated), provided with a rider beam, divided into $\frac{1}{10}$ milligram, and a set of weights ranging from 1 milligram to 100 grams; one large and one small desiccator; several air-tight boxes with screw lids, for taking

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pulp samples; one large steam bath (which should be connected with a still); one hot-air bath and one water bath for slow evaporation; porcelain dishes, sizes 4 and 6 inches; measuring flasks of the following capacities: 1,000, 500, 250, 100, 50, and 20 cubic centimeters, when filled to mark on neck; plain ungraduated flasks of the following capacities: 1,000, 500, 250, and 100 cubic centimeters; several 50-cubic-centimeter burettes, graduated in cubic centimeters and tenths of cubic centimeters, each provided with stand; set of pipettes, including the following sizes: 1, 2, 5, 10, 25, 50, and 100 cubic centimeters; set of graduated cylinders of the following capacities: 100, 500, and 1,000 cubic centimeters (preferably glass-stoppered for mixing standards); several ungraduated cylinders of about 200 cubic centimeters capacity; beakers, lipped Griffin shape, from Nos. 1 to 6; watch glasses of various sizes and a pair of ground glasses and clips; one iron, one large porcelain, one small porcelain, and one agate mortar, with pestle; funnels of various sizes; separating funnel; glass and rubber tubing, various sizes; platinum dish 50 cubic centimeters capacity; platinum crucibles, 25 and 15 grams respectively; platinum triangles for crucibles, and light and heavy platinum wire; ring-lamp stands and Bunsen burners; test tubes, various sizes; porcelain crucibles, various sizes; condenser for distilling liquids; filter paper, various sizes; filtering stand; set of reagent bottles; sample bottles, various sizes; several liter bottles for standard solution; wash bottle; drying apparatus and train for CO_2 determinations; thermometers, 212°F. and 200° C.; short-stem Baumé hydrometers of following ranges: 3° to 4°, 0° to 5°, 0° to 8°, 0° to 15°, and 15° to 40°; stirring rods, forceps, spatulas, and pinch cocks.

3. Hydrometers.—The hydrometer, whether it be on a Twaddell or Baumé scale, is very useful about a paper mill. It is used in all departments of the mill for the efficient regulation of the strengths of solutions used. The reading of the hydrometer is affected by the temperature, an increase in temperature decreasing the density of solutions, and it is therefore customary to add 1° Baumé for each 30° of temperature over 60° F. This allowance will only hold good for temperatures up to about 150° F. A greater allowance should be made for temperatures over this, about 4½° Baumé being allowed for a temperature of about 185° F.

4. **Drying Ovens.**—There are several kinds of drying ovens, some of which are filled with water and heated by means of a gas burner under the bath; but when one has to depend on gas supplied by a gas company, there are times when it is shut off and the use of the bath crippled.

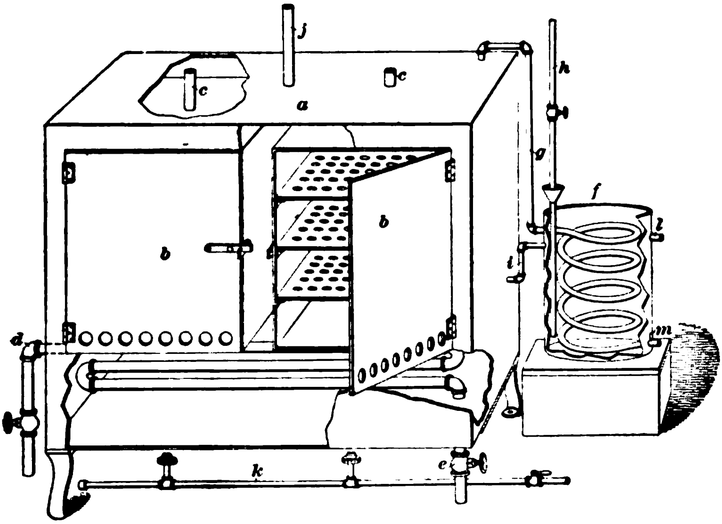

Fig. 1

The bath shown in Fig. 1 has been arranged and used by us, and has proved on all occasions to be a very convenient form of drying oven. It consists of a large copper bath *a*, which has two drying compartments *b* closed by means of doors perforated at the bottom, each having a chimney *c* extending to the outside, to permit the free passage of air through the bath. There are perforated shelves in each compartment, which can be removed if desired. The compartments are so arranged that steam circulates all around them. The bath is connected with the still *f*, which serves as a feed for it by means of the small connecting pipe *i*, the water in the bath being kept at a constant level with the overflow pipe *l* of the still. The pipe *g* comes from the top of the bath and connects with the worm of the still, the distilled water escaping at *m*. The cooling water is furnished to the still through pipe *h*. The still is at such a height that the bath is always half full of water, which is kept at a slow boil by means of the steam coil which enters at *d* and passes out at *e*. A pipe *j* extends into the boiler between the two compartments, and is provided with a valve, which when open stops the preparation of distilled water. As a precaution against the steam being turned off, a gas line *k* provided with two burners is run under the bath.

5. Knöfler Oven.—This oven is so arranged that the sample to be tested is suspended on a balance beam and lowered by means of a wire cage into the drying compartments, and the weight of the pulp can be read off at any time. It is convenient in that it is easy to determine when the pulp has become quite dry.

6. Chemicals.—The following is a list of the chemicals necessary for all analytical tests given in the following pages. All chemicals used should be chemically pure.

Hydrochloric acid
Sulphuric acid
Nitric acid
Acetic acid
Oxalic acid
Arsenious acid
Sulphanilic acid
Alcohol
Aniline sulphate
Ammonium oxalate
Ammonium phosphate
Ammonium sulphide
Ammonium chloride
Ammonium sulphocyanide

Barium hydrate
Barium chloride
Barium peroxide
Bromine
Calcium chloride
Ether
Carbon bisulphide
Iron perchloride
Iron protosulphate
Iron piano wire
Iron sulphide
Lead acetate
Lead peroxide
Mercury

Methyl orange
Manganese chloride
Marble chips
Phenol
Platinic chloride
Phloroglucine
Phenol phthalein
Potassium iodide
Potassium ferrocyanide
Potassium permanganate
Potassium bisulphate
Potassium chromate
Potassium sulphate
Potassium sulphocyanide
Potassium chlorate
Potassium hydrate
Sodium sulphate
Sodium phosphate
Sodium carbonate (deg.)
Sodium chloride
Sodium sulphite
Sodium hydrate
Sodium acetate
Sodium nitrate
Soda lime
Silver nitrate
Starch
Zinc sulphate
Zinc chloride
Zinc oxide
Zinc shot

7. Standard Solutions.—The following standard solutions will be found necessary:

Normal sulphuric acid
Normal hydrochloric acid
Normal oxalic acid
Normal sodium hydrate
Normal sodium carbonate
$\frac{n}{10}$ sulphuric acid
$\frac{n}{10}$ sodium hydrate
$\frac{n}{10}$ sodium arsenite
$\frac{n}{10}$ iodine
$\frac{n}{10}$ sodium chloride
$\frac{n}{10}$ silver nitrate
Standard potassium permanganate
Standard size

NOTE.—$\frac{n}{10}$ means tenth normal.

8. The above standard solutions, used in volumetric analysis, are made up in the following manner:

Normal Sulphuric Acid.—This solution should be made up very carefully, as it will furnish a good standard, by the aid of which all the other standard acids and alkalies can be made up. Take 30 cubic centimeters H_2SO_4 of, about 1.84 sp. gr. and mix with about 200 cubic centimeters of water. After cooling, transfer to a graduated mixing cylinder and dilute to 1 liter. Two samples are taken to standardize or rectify this standard. They should be measured carefully from a clean burette that has first been rinsed with some of this acid. Take the two samples, one of 10 cubic centimeters and the other of 20 cubic centimeters, and dilute to about 250 cubic centimeters in separate beakers. Bring to a boil, and, while stirring, precipitate with a $BaCl_2$ solution containing 1 gram $BaCl_2$ in 10 cubic centimeters of water, taking care that all of the sulphuric acid is precipitated. Allow this to settle for about 2 hours, filter, wash well with hot water, dry, ignite, and weigh as $BaSO_4$. The amount of H_2SO_4 in each sample is found, the two tests averaged, and the weight of H_2SO_4 in grams in 1 cubic centimeter of the acid solution calculated. This divided into 49 will give the number of cubic centimeters required to make 1 liter of the normal acid. In practical work, it will be found that one cannot spend much time in making up standard solutions, and it is therefore advisable to save this well-rectified standard and use it for no other purpose than that of checking other standards.

Tenth Normal $\left(\frac{n}{10}\right)$ *Sulphuric Acid.*—This can be prepared by measuring 100 cubic centimeters of the normal H_2SO_4 in an accurately graduated pipette that has been well cleaned and rinsed with the acid. The contents of the pipette are carefully rinsed into a liter flask and diluted to the mark. This standard can be rectified, if desired, in same way as above.

Normal Sodium Carbonate.—Weigh out about 53 grams of dry sodium carbonate, dissolve in about 500 cubic centimeters of hot water, cool, transfer to a liter flask, and dilute to the mark. Match this against the normal acid, using methyl orange as an indicator.

Normal Sodium Hydrate.—Dissolve 50 grams of $NaOH$ and dilute to 1 liter. Match against the normal acid, using methyl orange as an indicator. Dilute, if necessary, until

1 cubic centimeter is just equivalent to 1 cubic centimeter of normal acid.

Tenth Normal $\left(\frac{n}{10}\right)$ *Sodium Hydrate.*—Carefully measure 100 cubic centimeters of the normal solution and dilute to 1 liter. This can be verified by matching against $\frac{n}{10}$ sulphuric acid.

Normal Hydrochloric Acid.—Take 100 cubic centimeters of HCl (1.2 sp. gr.), dilute to 1 liter, and match against normal soda solution, using methyl orange as an indicator. It can be further rectified by precipitating with silver nitrate and calculating the number of cubic centimeters necessary to make 1 liter of strictly normal acid, as in the rectification of normal sulphuric acid.

Normal Oxalic Acid.—Dissolve 63 grams of pure crystals of oxalic acid in distilled water and dilute almost to 1 liter. This normal solution may be rectified by matching against a strictly normal alkali. It should be kept in a dark place.

Tenth Normal $\left(\frac{n}{10}\right)$ *Sodium-Arsenite Solution.*—There is no difficulty in preparing a good standard, provided good, chemically pure, dry, powdered arsenious acid As_2O_3 be used. Dissolve 12 grams of dry sodium carbonate in 100 cubic centimeters of hot water and add 4.95 grams (accurately weighed) of the arsenious acid. Cover the beaker with a watch glass and allow it to remain almost at a boil, stirring from time to time, until all the arsenious acid is dissolved, which requires from 1 to 1½ hours. (The above-mentioned proportions should be well observed, as any variation may cause considerable trouble in getting the arsenious acid into solution.) Cool, transfer to a liter flask, and dilute to the mark.

Tenth Normal $\left(\frac{n}{10}\right)$ *Iodine Solution.*—This is prepared by dissolving 12.7 grams of iodine with 18 grams of potassium iodide in 250 cubic centimeters of distilled water. This should be done in a glass-stoppered flask, as it requires much shaking in order to dissolve the iodine. The flask should not be heated. When dissolved, the iodine solution is transferred to a liter flask and diluted almost to the mark. In order to rectify this $\frac{n}{10}$ solution, it is matched against the $\frac{n}{10}$ sodium arsenite, using starch as an indicator.

Standard Potassium-Permanganate Solution. — Dissolve about 3 grams of pure crystals of potassium permanganate in 1 liter of distilled water. The value of this solution in term of iron is determined by titrating a solution containing a known quantity of iron. This can be done by the use of a solution of piano wire, which is known to contain 99.6 per cent. iron. Take a small flask of about 250 cubic centimeters capacity and fill one-third full of dilute sulphuric acid (1 volume of acid to 3 volumes of water), then add a few grains of sodium-carbonate crystals. Weigh accurately .2 gram of the above-mentioned wire into a flask, close with a rubber stopper provided with a valve, as shown in Fig. 2, and gently heat. When the wire is all dissolved, the flask is cooled as rapidly as possible, the contents rinsed into a large beaker partly filled with recently boiled distilled water. It is now diluted to about 500 cubic centimeters and titrated with the permanganate solution until the faint pink tint is permanent. Since the iron wire is only 99.6 per cent. pure, there has been dissolved in reality only .2 × .996 or .1992 gram of pure iron. If it requires 25 cubic centimeters of permanganate, 1 cubic centimeter will be equivalent to .1992 ÷ 25, or .007968 gram of iron.

Standard Size Solution.—Standard size solution is prepared as follows: Dissolve 20 to 25 grams of good heavy rosin size in about 250 cubic centimeters of 95-per-cent. alcohol, filter, and dilute almost to 1 liter with a 60-per-cent. alcohol solution. Add a little phenol phthalein and then add a solution of caustic soda, drop by drop, from a burette, shaking after each addition, until a faint pink color appears. This neutral size solution is used as the standard after first determining its value. The value is determined by means of pure crystallized potassium alum, 1 part of which precipitates 2.36 parts of neutral rosin size. Weigh out 10 grams of the crystals that have previously been pressed between two filter papers, dissolve, and dilute to 1 liter. One cubic centimeter will then contain .01 gram of alum. A flask of about 200 cubic centimeters capacity is then filled about two-thirds full of distilled water and 20 cubic centimeters of this size solution run in from a burette. The alum solution is then run in from another burette, a few drops at a time. The flask is then closed with a stopper, shaken vigorously, and allowed to stand after each addition until the precipitate rises to the top, which it will do very rapidly. This is continued until the solution is left perfectly clear, after the precipitate has risen. The number of cubic centimeters of alum used multiplied by .01 gives the amount of alum in grams required to precipitate 20 cubic centimeters of standard size, which multiplied by 2.36 gives the number of grams of neutral size in 20 cubic centimeters of this solution. If 10 cubic centimeters of the alum solution be used, 20 cubic centimeters of the standard size contains .236 gram of neutral rosin size.

Tenth Normal $\left(\frac{n}{10}\right)$ *Sodium-Chloride Solution.*—This is prepared by accurately weighing 5.837 grams of chemically pure sodium chloride (which has been previously heated in a covered platinum crucible to a low, red heat for some minutes and cooled in a desiccator), dissolving in distilled water, and making up to 1 liter. It is always advisable to keep this standard solution on hand to assist in making up $\frac{n}{10}$ silver-nitrate solutions.

Tenth Normal $\left(\frac{n}{10}\right)$ *Silver-Nitrate Solution.*—This solution is prepared by weighing out 17 grams of pure crystallized silver nitrate, dissolving and diluting to almost 1 liter. It is then matched against the $\frac{n}{10}$ sodium-chloride solution, using potassium chromate as an indicator, and adjusted to exact

$\frac{n}{10}$ solution. One cubic centimeter of this solution will then be equivalent to .00355 gram of chlorine or .00585 gram of sodium chloride.

9. Indicators.—The following indicators will be found necessary:

Phenol phthalein is soluble in 50-per-cent. alcohol, and is made up for use as follows: Take a mixture of 105 cubic centimeters of alcohol (95 per cent.) and 95 cubic centimeters of water. Add to this 2 grams of phenol phthalein and stir until all is dissolved. A few drops of this indicator are sufficient for titrating. It will produce no color in acid liquids, but the slightest trace of caustic alkali will change it to purple red. This indicator can be used in alcoholic solutions and is useful in titrating organic acids.

Methyl Orange.—If methyl orange contains impurities that are not soluble in water, it should be recrystallized from alcohol. It should not be used for titrating organic acids. A convenient strength is .1 gram to 100 cubic centimeters of water. One or two drops is sufficient for 100 to 150 cubic centimeters of the solution titrated. A good end reaction cannot be obtained if too much of the indicator is used.

Starch is prepared as follows: About 300 cubic centimeters of water is brought to a boil in a flask of about 1 liter capacity. Four grams of starch is then mixed in a little water until all the lumps are broken and it forms a thin, pasty mass, which is added to the boiling water, and the whole allowed to boil for 5 minutes. A mixture of 5 grams of zinc chloride in a little water is then added, and the whole allowed to boil 1 minute longer. It is then removed from the flame, diluted to about 900 cubic centimeters, well mixed, and allowed to stand over night. The clear starch solution is then decanted into a bottle and is ready for use.

Iodized Starch.—This is made by taking some of the above starch solution and adding a small amount of a solution of potassium iodide.

Potassium Chromate.—A convenient strength is 1 gram to 10 cubic centimeters of water. A few drops of the solution are sufficient for 100 to 150 cubic centimeters of the solution to be titrated.

ANALYTICAL METHODS

10. Analysis of Soda Ash.—The usual determination made in this substance is for sodium oxide. If it falls below the requirement in this, free caustic soda and sulphuric acid are determined. The material should first be carefully sampled. If it is received in bags, a portion should be taken from each of seven or eight bags selected at random from each car and thoroughly mixed and cut down in size if necessary.

Determination of Sodium Oxide.—Weigh out 15.5 grams of the sample, dissolve in about 400 cubic centimeters of hot water, let cool, wash into a liter flask, and dilute to the mark. Measure out 100 cubic centimeters of this solution into a beaker by means of a pipette, add two drops of methol orange, and titrate with a normal acid. The number of cubic centimeters of the normal acid used multiplied by 2 gives the percentage of sodium oxide in the sample. Good soda ash should contain 58 per cent. of sodium oxide.

Determination of Free Caustic Soda.—If it is desired to test for caustic soda, measure out 100 cubic centimeters of the original solution and add a slight excess of *neutral* barium chloride, to precipitate all the carbonate. Filter off the precipitated $BaCO_3$ and titrate the filtrate with normal acid, using phenol phthalein as an indicator.

Determination of Sulphuric Acid.—Take 100 cubic centimeters of the solution, equivalent to 1.55 grams of ash, acidify carefully with hydrochloric acid, bring to a boil, and precipitate the H_2SO_4 as $BaSO_4$ by means of barium chloride. Let stand in a warm place for about 2 hours, to settle the precipitate. Decant the clear liquid through an ashless filter paper, then wash the precipitate upon it, and wash thoroughly with hot water. Dry, ignite, and weigh as $BaSO_4$. The weight of the $BaSO_4$ multiplied by the factor .34335 gives the weight of the SO_3, which multiplied by 100 and divided by 1.55, the weight of ash taken, gives the percentage of SO_3 in the ash. This is calculated to Na_2SO_4, according to the following proportion:

SO_3 : Na_2SO_4 = percentage of SO_3 : percentage of Na_2SO_4;
or 80 : 142 = percentage of SO_3 : x,
where x = percentage of Na_2SO_4.

11. Analysis of Causticizing Lime.—This lime is used for converting the carbonate of soda into caustic soda. The caustic lime when added to the carbonate solution is first converted into $Ca(OH)_2$, which reacts with the sodium carbonate, forming caustic soda, and calcium carbonate, which settles to the bottom of the tank as *lime sludge*. A good lime for this purpose should be well burned, as free as possible from impurities, and contain a high percentage of CaO. Lime that is slaked when received is not suitable for causticizing, as more of it is required to do the same work than if unslaked. In analyzing lime ordinarily, calcium oxide, carbon dioxide, and water only are determined.

Sampling the Lime.—A very large sample should be taken from different parts of the car and the whole broken up into lumps about the size of a pea. It should then be mixed thoroughly and quartered several times, to reduce to a small sample. This sample should be ground very fine, well mixed, and kept in a tightly corked bottle.

Determination of Calcium Oxide.—Weigh out 10 grams of the sample, slake, and wash carefully into a 500-cubic-centimeter flask, filling it to the mark. Take 50 cubic centimeters of the clear solution, which is equivalent to 1 gram of lime, wash into a beaker, and titrate with normal oxalic acid, using phenol phthalein as an indicator. The number of cubic centimeters of normal acid used multiplied by 2.8 gives the percentage of CaO in the sample, which includes also the magnesium oxide, but as this is usually very low in this lime, its presence can for all practical purposes be disregarded.

Determination of Carbon Dioxide.—This can be very accurately determined by treating a carefully weighed sample in a flask with hydrochloric acid and absorbing the liberated CO_2 in soda lime. This method, however, necessitates the use of a drying apparatus for the air that passes through the flask and also a train of U tubes with an aspirating bottle at the end of the train. For all practical purposes, the CO_2 may be determined as follows:

Take 50 cubic centimeters of the solution used for the calcium-oxide determination after shaking, add from a burette an excess of normal hydrochloric acid, and titrate the excess of acid with normal sodium hydrate. The number of cubic centimeters of hydrochloric acid used less the number of cubic centimeters in excess, as found by the sodium-hydrate titration, gives the total number of cubic centimeters neutralized by the CaO and $CaCO_3$. The number of cubic centimeters thus found less the number of cubic centimeters of normal oxalic acid used in the estimation of calcium oxide gives the number of cubic centimeters of hydrochloric acid used in titrating the $CaCO_3$. This number multiplied by 5 gives the percentage of $CaCO_3$ in the sample. To find the percentage of CO_2, multiply the percentage of $CaCO_3$ by .44.

Determination of Water.—This can be determined by weighing 1 gram of the lime sample into a platinum crucible, gently heating at first, and gradually raising the temperature to a strong, red heat. Cool in a desiccator and weigh quickly. Repeat until a constant weight is obtained. The loss in weight will be due to water and CO_2. Determine the CO_2 as above. Subtract the weight of the CO_2 in 1 gram from the total loss of weight on heating. The difference will be the loss due to water. This weight multiplied by 100 gives the percentage of water in the sample.

12. Complete Analysis of Lime.—When a complete analysis of lime is desired, the following determinations are usually made—viz.: silica, sesquioxides of iron and aluminum, calcium oxide, magnesium oxide, sulphuric acid, and carbon dioxide.

Determination of Silica.—Weigh out 2 grams of the carefully prepared sample and fuse in a platinum crucible with about 8 grams of sodium carbonate. Dissolve the fused mass with hot water in a porcelain dish with the use of a slight excess of dilute hydrochloric acid, keeping it well covered with a watch glass. Boil and evaporate to dryness. Moisten thoroughly with strong hydrochloric acid and again evaporate to complete dryness. Take up in dilute hydrochloric acid, boil, and filter through an ashless filter, using a "policeman," or rubber-tipped glass rod, to remove the fine particles of silica from the dish. Wash thoroughly with hot water. The filtrate and washing should be collected in a 200-cubic-centimeter flask, cooled, and filled to the mark. The filter paper and residue are transferred to a crucible, ignited, and weighed. The weight of the residue multiplied by 50 gives the percentage of SiO_2 in the sample.

Determination of Sesquioxides of Iron and Aluminum. Take 100 cubic centimeters of the filtrate from the silica determination, heat nearly to boiling, add a few drops of nitric acid, and precipitate the iron and aluminum with a slight excess of ammonium hydrate. Then take up again in a slight excess of hydrochloric acid and reprecipitate with a slight excess of ammonium hydrate, bring to a boil, and allow to boil gently until the odor of ammonia is no longer perceptible. Allow the precipitate to settle, decant the clear liquid through an ashless filter, wash several times by decantation, boiling with water each time, and finally collect the precipitate on the filter. Dry in an oven, transfer to a weighed crucible, ignite, and weigh as $Al_2O_3 + Fe_2O_3$. This weight multiplied by 100 gives the percentage of these mixed oxides in the sample.

Determination of Calcium Oxide.—Heat the filtrate from the previous analysis to boiling and add a slight excess of ammonium-oxalate solution, stirring for some minutes. Allow this to settle for about 3 hours, wash well with water containing ammonia (25 cubic centimeters of strong ammonia to 175 cubic centimeters of water), and dry. Transfer the precipitate to a platinum crucible, burn the filter paper, and add its ash. Treat with a few drops of dilute sulphuric acid, heat gently, to drive off the excess of H_2SO_4, then heat to redness, cool in desiccator, and weigh as calcium sulphate. This weight multiplied by .412 × 100 gives the percentage of CaO in the sample.

Instead of treating the precipitate with H_2SO_4, it may be converted directly into CaO by igniting to constant weight, which requires a long treatment at a very high temperature. This weight multiplied by 100 gives the percentage of CaO.

Determination of Magnesium Oxide.—To the filtrate from the calcium-oxide determination, which should not exceed 200 cubic centimeters, add a little NH_4OH and precipitate while stirring with sodium phosphate. Allow this to settle for about 2 hours, filter, and wash with ammonia water of the same strength as was used for washing the calcium oxalate. Dry, transfer the precipitate to a porcelain crucible, burn the filter, and add its ash. Then ignite to constant weight and weigh as magnesium pyrophosphate $Mg_2P_2O_7$. This weight multiplied by .36036 × 100 gives the percentage of MgO in the sample.

Determination of Sulphuric Acid.—Take the remaining 100 cubic centimeters of the filtrate from the silica determination, heat to boiling, and precipitate the sulphuric acid as $BaSO_4$ with barium chloride. Let stand in a warm place for about 2 hours, or until the precipitate is settled. Decant the clear liquid through an ashless filter, wash twice by decantation with hot water, and finally on the filter with hot water until a few drops of the filtrate give no test for chlorides with silver nitrate. Dry, transfer to a crucible, burn the filter as before, and ignite to constant weight. The weight of the $BaSO_4$ multiplied by .34335 × 100 gives the percentage of SO_3 in the sample.

Determination of Carbon Dioxide. — This determination should be made as described in Art. **11.**

The following analysis shows the composition of a good causticizing lime:

Silica SiO_2	.62%
$Fe_2O_3 + Al_2O_3$	.84%
Calcium oxide CaO	95.91%
Magnesium oxide MgO	.51%
Carbon dioxide CO_2	.92%
Water	1.15%
Undetermined	.05%
Total	100.00%

13. Analysis of Magnesia Lime.—This lime is used for making up bisulphite liquors for the sulphite process. It should be well burned, contain high percentages of both calcium and magnesium oxides, and be fairly free from impurities. The same determinations are made as in cauticizing lime and the same methods used.

In the determination of calcium and magnesium oxides, owing to the fact that some magnesium oxalate will be precipitated with the calcium oxalate, the following precautions must be taken: Precipitate the calcium oxalate in the usual manner, and after slight washing redissolve the precipitate in hydrochloric acid, make ammoniacal, reprecipitate the calcium, filter, wash, and treat as calcium oxalate, as previously described. The filtrates are combined, the whole evaporated to 200 cubic centimeters, and the magnesium precipitated with sodium phosphate, as in the preceding article.

The following is an analysis of a good magnesia lime for the sulphite process:

Silica and insoluble	.22%
$Fe_2O_3 + Al_2O_3$	.19%
Calcium oxide CaO	50.92%
Magnesium oxide MgO	44.32%
SO_3	1.51%
Water	1.23%
Carbon dioxide CO_2	1.48%
Undetermined	.13%
Total	100.00%

14. Analysis of Sludge from Causticizing Pans. This is tested in order to determine the loss in per cent. of soda, etc. at this point. After the liquor from the third wash has been run off as low as possible, the sludge is well agitated and a sample taken. The determinations usually made are insoluble matter, sodium oxide, calcium oxide, calcium carbonate, and moisture.

Determination of Insoluble Matter.—Weigh out 20 grams, or more if necessary, of the sample in a small beaker. Transfer to a porcelain dish and take up with an excess of hydrochloric acid and evaporate to dryness to expel all HCl. Take up again in hydrochloric acid and water, boil, filter, and wash the residue thoroughly with hot water. Ignite in a crucible to constant weight. This weight multiplied by 100 and divided by the weight of the sample taken gives the percentage of insoluble matter.

Determination of Sodium Oxide.—Weigh out approximately 45 or 50 grams of the sample, transfer to a porcelain dish, and evaporate to dryness with a little ammonium carbonate. Take up with water and repeat this evaporation with ammonium carbonate. Take up again in hot water and allow to remain almost at a boil for some time. Filter into a 500-cubic-centimeter flask, wash thoroughly with hot water, cool filtrate and washings, and fill up to the mark. Take 100 cubic centimeters of this filtrate and titrate with a normal acid, using methyl orange as an indicator. The number of cubic centimeters of acid required multiplied by .031 gives the weight in grams of the sodium oxide in 100 cubic centimeters of the solution. This weight multiplied by 5 × 100 and divided by the weight of sludge used in making the sample solution gives the percentage of Na_2O in the sludge.

Determination of Caustic Lime.—Take 100 cubic centimeters of the sludge, weigh in a 100-cubic-centimeter flask, wash into a beaker, and titrate with normal oxalic acid, using phenol phthalein as an indicator. The number of cubic centimeters required less the number of cubic centimeters neutralized by the Na_2O, as calculated for the weight of sludge used from the data obtained in the sodium-oxide determination, is the number of cubic centimeters of normal oxalic acid neutralized by the CaO. This number multiplied by .028 (the value of 1 cubic centimeter of normal oxalic acid in lime) × 100 and divided by the weight of the sludge taken gives the percentage of calcium oxide or caustic lime.

Determination of Calcium Carbonate. — Take about 10 grams of the sludge, after agitating well, and titrate with normal hydrochloric acid, using methyl orange as an indicator. The number of cubic centimeters of acid used, less the number required for sodium oxide and for caustic lime as previously determined for the same weight of sample, gives the number of cubic centimeters of normal acid neutralized by the calcium carbonate. This number multiplied by .05 gives the weight of calcium carbonate in the sample taken. This weight multiplied by 100 and divided by the weight of sludge taken gives the percentage of calcium carbonate.

Determination of Moisture.—This determination is made by weighing out about 50 grams of the sample as quickly as possible and drying at 100° C. to constant weight. The loss in weight multiplied by 100 and divided by the weight of sample taken gives the percentage of moisture. Other constituents of sludge may be determined by methods given in Art. **12.**

The following is an analysis of a lime sludge:

Moisture	76.780%
Silica SiO_2	.090%
$Fe_2O_3 + Al_2O_3$	.250%
Magnesium oxide MgO	.085%
Sodium oxide Na_2O	.350%
Calcium hydrate $Ca(OH)_2$	1.070%
Calcium carbonate $CaCO_3$	21.290%
Undetermined	.085%
Total	100.000%

15. Analysis of Bleaching Powder.—The value of bleaching powder depends on the percentage of available chlorine in it. On standing it loses its strength, especially if kept in a damp place. A good bleaching powder should contain from 36 to 37 per cent. of available chlorine, but it is generally accepted if it tests over 35 per cent.

Sampling.—In order to get a fair sample from a car, small samples should be taken from at least five casks. This is best done by means of a long, half-inch copper tube, which is thrust into the cask after boring a hole in it, withdrawn, and the contents transferred to the sample bottle by gently tapping the tube. The sample bottle should be perfectly dry and kept closed as much as possible.

Determination of Available Chlorine.—Weigh out 7.1 grams of the well-mixed sample, transfer to a porcelain mortar, add a little water, and grind with the pestle, taking care to avoid loss. Allow to settle for a moment and decant into a liter flask. Repeat this three times and finally wash the contents of the mortar into the flask, using a clean finger to rub off the mortar and pestle. Fill to the mark with water, shake well, and transfer 50 cubic centimeters to a beaker by means of a pipette. Titrate with $\frac{n}{10}$ sodium-arsenite solution, stirring while adding the standard. The end point is reached when a drop of the solution no longer gives the blue coloration to iodized starch paper (this paper is prepared by soaking strips of filter paper in iodized starch solution). When the above quantity of sample is used, each cubic centimeter of the standard used is equivalent to 1 per cent. of available chlorine. With practice, this test can be made rapidly with good results.

The bleaching powder should be tested as soon as possible after sampling, and after starting to test there should be no delay, as otherwise the results will come too low.

Determination of Chlorate.—Sometimes it is desired to determine the amount of chlorate present in bleaching powder. For all practical purposes, this can be done by adding a little dilute sulphuric acid to the sample that has been titrated for available chlorine and rapidly titrating again, thus determining the chlorine present as chlorate.

The following is a complete analysis of bleaching powder:

Available chlorine	36.80%
Chlorine as chlorate	.22%
Chlorine as chloride	.42%
Lime CaO	43.98%
Magnesia MgO	.39%
$Fe_2O_3 + Al_2O_3$	.41%
Water (by difference)	17.78%
Total	100.00%

16. Analysis of Bleach Sludge.—It is important that this should be tested from time to time, to ascertain the loss of available chlorine in the dump. The sludge, after drawing off the wash as closely as possible, should be agitated and the sample taken.

Determination of Available Chlorine.—Measure 50 cubic centimeters of the well-mixed sample into a beaker by means of a pipette, which is rinsed out into the beaker. Titrate with $\frac{n}{10}$ sodium arsenite as before. The number of cubic centimeters used multiplied by .071 gives the number of grams of available chlorine per liter of sludge, which multiplied by 3.785 (the number of liters in 1 gallon) gives the weight of available chlorine in 1 gallon. This multiplied by 3 gives the approximate loss of bleaching powder in 1 gallon, and, knowing the cubical contents of the tank and the number of inches dumped, the loss in the dump can be easily calculated.

In a bleach that settles well, this loss ranges from .6 per cent. to 1 per cent. of the bleach mixed. If the bleach does not settle well, the loss will reach 3 per cent. and may go even higher.

17. Analysis of Black Ash.—Black ash is the ash that is recovered after the liquor used in the soda process, in which the wood has been cooked, has been evaporated and burned.

Preparation of Sample.—Quite a large sample should be taken, ground up, mixed, and quartered several times. The last portion should be ground very fine and well mixed in the sample bottle. Weigh out 50 grams of this carefully prepared sample and boil up with water. Stir well from time to time for at least 1 hour, filter into a liter flask, wash well, cool, and dilute to the mark. It is customary to make determinations of caustic soda, sodium sulphide, sodium carbonate, sodium sulphate, and sodium chloride.

Determination of Caustic Soda.—Take 50 cubic centimeters of the clear liquid and precipitate the carbonate with a slight excess of barium-chloride solution in a 200-cubic-centimeter flask, add hot water, shake well, cool, fill to the mark, and after again shaking, allow to settle. Take 100 cubic centimeters of the clear portion and titrate with normal acid, using methyl orange as an indicator. Multiplying the number of cubic centimeters of acid used by 4 and dividing by 1.25 gives the percentage of $NaOH$ in the ash. This also includes any Na_2S present, and if it is considerable, the number of cubic centimeters of standard acid neutralized by it must be deducted from the whole before the calculation of the caustic soda is made.

Determination of Sodium Sulphide.—Take 25 cubic centimeters of the clear liquor, equivalent to 1.25 grams of the ash, dilute to 200 cubic centimeters with recently boiled water, acidify with acetic acid, and titrate quickly with $\frac{n}{10}$ iodine solution, using starch as an indicator. The number of cubic centimeters of standard used, multiplied by .39 and divided by 1.25, gives the percentage of Na_2S in the ash.

Determination of Sodium Carbonate.—Take 25 cubic centimeters of the original clear liquor and titrate with normal hydrochloric acid, using methyl orange as an indicator. The number of cubic centimeters of acid used less the number of cubic centimeters neutralized by the caustic soda, as determined for the same volume of liquor, multiplied by 5.3 and divided by 1.25, gives the percentage of Na_2CO_3 in the ash.

Determination of Sodium Chloride.—Take 25 cubic centimeters of the clear liquor, neutralize with nitric acid, and boil to expel hydrogen sulphide. Filter, wash, cool, add a little potassium-chromate indicator, and titrate with $\frac{n}{10}$ silver-nitrate solution. The number of cubic centimeters of silver nitrate required multiplied by .585 and divided by 1.25 gives the percentage of sodium chloride in the ash.

Determination of Sodium Sulphate.—This can be determined by dissolving 25 grams of ash in hot water, filtering and washing well, acidifying filtrate slightly with hydrochloric acid, boiling to expel all CO_2, and precipitating with a slight excess of barium-chloride solution. Let stand in a warm place until settled, filter, and wash thoroughly with hot water. Dry, ignite in a crucible, and weigh as $BaSO_4$. Calculate to Na_2SO_4 by multiplying by .6094. The weight of Na_2SO_4 multiplied by 4 gives its percentage in the ash.

The following is an analysis of a sample of black ash:

Sodium carbonate	78.15%
Sodium hydrate	1.83%
Sodium sulphate	2.55%
Sodium sulphide	.50%
Sodium chloride	6.60%
Carbon	4.30%
Silica	5.10%
Calcium carbonate	.72%
Undetermined (difference)	.25%
Total	100.00%

18. Analysis of Alum.—In the paper industry the term **alum** is applied to sulphate of aluminum $Al_2(SO_4)_3$, $18H_2O$, which, strictly speaking, is not an alum. The analysis of an alum is quite long and complicated, but if carried on with care very good results can be obtained. The following determinations and tests are usually made:

Determination of Water.—Weigh out 2 grams of the well-mixed sample into a platinum crucible. Heat gradually until a low, red heat is reached and allow to remain at this temperature until fumes of SO_3 are perceptible. Cool in a desiccator and weigh. The loss of weight gives the amount of water together with some SO_3. The loss of SO_3 may be determined as follows:

Add about 10 cubic centimeters of hot, concentrated hydrochloric acid to the contents of the crucible and allow to stand in a warm place for about half an hour, when the lumps will all be broken down, provided the heating was not too high or too long, when driving off the water. The contents of the crucible are now washed into a small beaker with hot water, diluted to about 50 cubic centimeters, and heated until all is dissolved excepting what little insoluble matter there may be in the alum. Filter and wash well with hot water. Precipitate the sulphuric acid in the filtrate with barium chloride, as in the determination of sulphuric acid described in Art. **10.** Filter, wash well with hot water, dry, and ignite to constant weight. The weight of $BaSO_4$ multiplied by .34335 gives the weight of SO_3. This weight subtracted from the weight of the total SO_3 in the same weight of sample, as found by a separate determination, gives the loss of weight due to SO_3. Subtracting this loss from the total loss on heating gives the loss due to water, which multiplied by 50 gives the percentage of water in the alum.

Determination of Insoluble Matter.—Weigh out 20 grams of the alum and dissolve in about 300 cubic centimeters of hot water. Filter through an ashless filter into a liter flask and wash well with hot water. Dry, ignite, and weigh the residue, the weight of which multiplied by 5 gives the percentage of insoluble matter in the alum. Cool the filtrate and dilute to the mark.

Determination of Alumina.—The alumina and ferric oxide are determined together. The ferric oxide is determined in a separate portion and subtracted from the mixed oxides, giving the alumina.

Take 50 cubic centimeters of the filtrate from the insoluble determination (equivalent to 1 gram of alum), dilute to 200 cubic centimeters, and precipitate with a slight excess of ammonium hydrate. Neutralize the excess of ammonia with hydrochloric acid and add NH_4OH gradually, drop by drop, until its odor can be detected in the solution. Bring almost to a boil and allow to remain so, after covering with a watch glass, until the odor of ammonia can no longer be detected. The volume of liquid should be kept as nearly constant as possible by additions of a little hot water. Boil, allow to settle, and decant the clear portion through an ashless filter. Boil up again with hot water, settle, and decant. Repeat this several times, finally wash the precipitate upon the filter, wash well with hot water, dry, ignite, and weigh as $Al_2O_3 + Fe_2O_3$.

The above analysis will hold good provided there is no zinc in the alum; but when zinc is present, which can be detected by precipitating with ammonium hydrate, filtering, and adding a few drops of yellow ammonium-sulphide solution, which will give a flocculent, white precipitate, the iron and aluminum oxides should be precipitated as basic acetates. Take about 50 cubic centimeters of the original solution and dilute to about 450 cubic centimeters, bring to a boil, and add about 2 grams of sodium acetate and a few drops of acetic acid. Boil the solution for about 15 minutes, allow to settle, and decant through an 11-centimeter ashless filter. Wash several times by decantation, bring to a boil each time, and finally wash on to the filter with hot water containing a little ammonium acetate. Evaporate the filtrate to about 150 or 200 cubic centimeters and filter again through a separate filter paper. Wash well and add to the first residue. Dry, ignite, and weigh as $Al_2O_3 + Fe_2O_3$.

Determination of Zinc.—The filtrate from the aluminum and ferric hydrates should be neutralized as nearly as possible, and the zinc precipitated as the sulphide with ammonium sulphide. The reagent should be added drop by drop, to avoid a large excess. Boil for about 20 minutes and allow to settle. Test with a drop of ammonium sulphide to make sure that all the zinc is precipitated, filter, wash, dry, and ignite to constant weight at a strong heat. This weight multiplied by 100 will give the percentage of zinc oxide in the alum.

Determination of Iron.—1. Take 10 grams of the alum, dissolve in water, filter into a flask that holds about 300 cubic centimeters, and dilute to about 150 cubic centimeters. Add 8 or 10 grams of granulated zinc and about 40 cubic centimeters of concentrated sulphuric acid. Close the flask with a valve made as shown in Fig. 2. This valve consists of a glass tube *d* passing through the cork *e*. On the end of this glass tube is slipped a rubber tube *b*, having a slit *c* for the escape of gas. The upper end of *b* is closed by a glass plug *a*. This allows the hydrogen generated to escape, but does not allow the air to enter. Set the flask on the hot plate until the zinc is dissolved. This will be sufficient to reduce all the ferric iron to the ferrous state. Boil for about a minute to drive out any remaining hydrogen, cool as quickly as possible, and transfer to a larger beaker. Dilute to about 500 cubic centimeters with distilled water that has been recently boiled and titrate with standard permanganate solution. The number of cubic centimeters used multiplied by the factor for the solution gives the number of grams of iron in 10 grams of alum. Calculate this to Fe_2O_3 by multiplying by 1.4285. The number of grams of Fe_2O_3 multiplied by 10 gives the percentage of Fe_2O_3 in the alum.

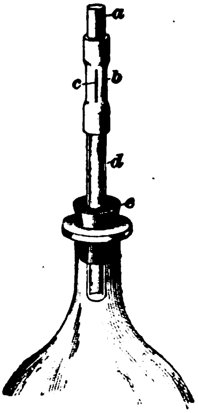

FIG. 2

2. The percentage of iron oxide can also be determined by dissolving 20 grams of alum in water, bringing to a boil, and adding a large excess of potassium-hydrate solution. The aluminum hydrate formed is dissolved by the *KOH*, while the ferric hydrate is not affected. Filter, wash, and

dissolve the precipitate in warm dilute hydrochloric acid. Heat to boiling and reprecipitate the iron with ammonium hydrate. This precipitate will now be free from alumina. Filter, washing first by decantation and finally on the filter paper, dry, ignite, and weigh as Fe_2O_3.

Determination of Sulphuric Acid.—Take 50 cubic centimeters of the prepared sample solution (equivalent to 1 gram of alum), dilute to about 200 cubic centimeters, acidify with hydrochloric acid, bring to a boil, and precipitate with barium chloride, and treat as in Art. **10.** The weight of the $BaSO_4$ multiplied by .34335 × 100 gives the percentage of SO_3 in the alum.

Determination of Free Acid.—It is very important in determining the value of an alum to know the amount of free acid present, as the same attacks the coloring matter and at the same time injures the machinery of the mill. Many methods have been suggested for the quick determination of the free acid, but they are all very unsatisfactory. Three methods are here given:

1. Digest a weighed quantity of alum for about 15 hours in strong alcohol. Filter, wash with the same strength alcohol, and titrate with $\frac{n}{10}$ alkali, using phenol phthalein as an indicator. Calculate the percentage of free acid.

2. Dissolve 2 grams of alum in 5 cubic centimeters of water and add 5 cubic centimeters of cold, saturated solution of ammonium sulphate, stirring thoroughly. Now add 50 cubic centimeters of 95-per-cent. alcohol, filter, evaporate the filtrate on a water bath, take up in water, and titrate with $\frac{n}{10}$ alkali, using phenol phthalein as an indicator.

3. The following is the most accurate method for the determination of the free acid in alum: First, make a complete analysis of the alum. Determine the amount of sulphuric acid necessary to combine with all the bases. If there be more acid than is necessary to combine with all the bases, the excess may be considered as free sulphuric

acid. If there is not acid enough to combine with all the bases, combine the acid with all the bases except alumina, and then use up all the latter possible, reporting the balance as basic alumina Al_2O_3.

Sizing Test.—The standard size is prepared as described in Art. **8.** Dissolve 10 grams of alum in water and dilute to 1 liter. Fill a 200-cubic-centimeter flask about two-thirds full of distilled water, and run in from a burette 20 cubic centimeters of size solution. Now, add the alum solution from a burette, a few drops at a time. Shake violently and allow the precipitate to rise after each addition. Towards the end, the alum should be added, a drop at a time. When the precipitate, on rising, leaves the solution perfectly clear, take the reading of the alum burette. The number of cubic centimeters of alum solution multiplied by .01 gives the number of grams of alum necessary to precipitate 20 cubic centimeters of standard size, and this, divided into the factor for neutral dry size in 20 cubic centimeters of standard size, gives the number of parts of neutral dry size precipitated by 1 part of alum.

The above is a good, quick test for the precipitating power of alum, but it does not give much of an idea as to its quality, as it may be a very acid alum and still have a good precipitating power.

Ultramarine Test. — This test in connection with the sizing test will give a fair idea as to the fitness of an alum for paper makers' use, provided, however, the alum contains a low percentage of iron.

Weigh out .2 gram of ultramarine, transfer to a 100-cubic-centimeter flask, and fill to the mark with distilled water. Shake well, in order to thoroughly mix the ultramarine, taking care that none remains on the bottom of the flask. Weigh out 2 grams of the alum to be tested, and also 2 grams of an alum that has already been well tested and with which the sample is to be compared. Dissolve each sample in 100 cubic centimeters of distilled water. Take 50 cubic centimeters of each of these and transfer to two properly labeled 100-cubic-centimeter flasks. Now, to each of the alum solutions add 10 cubic centimeters of the ultramarine mixture (which should first be well shaken). Allow them to stand for some time, with frequent shaking, and note the effect of the alum on the color of each mixture. This is but a comparative test as to the action of the alum on ultramarine.

Samples of alum analyses in which the different elements were determined and bases combined with acid are given in Table I.

19. Analysis of Agalite.—Agalite is a silicate of magnesia of a fibrous nature and is retained very well in the paper, the retention being from 60 to 90 per cent. of the amount used. It contains more grit than clay. It should not contain much soluble matter, as this cuts down its retention. In analyzing, the following determinations are usually made:

Determination of Water. —Weigh out 2 grams of the agalite in a platinum crucible and heat for some time at a red heat. Cool in a desiccator and weigh. Repeat until a constant weight is obtained. The loss of weight multiplied by 100 and divided by 2 gives the percentage of water in the sample.

Determination of Silica.—Fuse 1 gram of the agalite in a platinum crucible with 10 grams of a mixture of 1 part sodium carbonate and 1 part potassium carbonate. Cool the bottom of the crucible in cold water, which will cause the solidified mass to break away from the sides. Transfer with the aid of hot water to a 4-inch porcelain dish. Cover with a watch glass, add about 100 cubic centimeters of hot water, and then cautiously add hydrochloric acid until the mass is broken up and effervescence ceases; then proceed with the determination of silica as directed in Art. **12.** The weight of the silica multiplied by 100 gives its percentage in the agalite.

Determination of Sesquioxides of Iron and Aluminum. Treat the filtrate from the silica determination in exactly

TABLE I

Alum Sample	Water. %	Insol. %	$Al_2(SO_4)_3$ %	$FeSO_4$. %	$ZnSO_4$. %	Na_2SO_4. %	Free H_2SO_4. %	Basic Al_2O_3. %	Parts of Neutral Dry Size Precipitated by 1 Part Alum	Remarks
A	41.83	.170	49.18	.62	6.36			1.54	3.47	This alum is very good with ultramarine
B	41.65	.046	56.76	.68				.61	3.59	Fair with ultramarine
C	45.59	.035	53.49	.51			.25		3.51	Strong action on ultramarine
D	45.28	.060	53.26	.41			.81		3.53	Strong action on ultramarine
E	43.57	.047	54.98	.33				.85	3.58	Fair with ultramarine
F	44.18	.100	54.88	.32				.41	3.55	Slight action on ultramarine
G	43.16	.070	52.02	.38	3.68			.76	3.55	Fair with ultramarine
H	43.63	.076	51.83	.62	1.35	.7		1.24	3.41	Good with ultramarine
I	41.90	.600	54.29			3.0		.30	3.41	Slight action on ultramarine
J	42.32	.040	56.85	.25				.45	3.63	Slight action on ultramarine
K	43.29	.056	55.59	.57	.05			.19	3.49	Acts considerably upon ultramarine

the same way as in the determination of the sesquioxides of iron and aluminum in Art. **12.** The weights of the mixed oxides $Fe_2O_3 + Al_2O_3$ multiplied by 100 gives their percentage. Unless the presence of iron is very marked, it is unnecessary to separate the Al_2O_3 from the Fe_2O_3, but if desired, proceed as in Art. **18** in the determinations of alumina and iron.

Determination of Calcium Oxide.—Evaporate the filtrate from the previous determination to about 200 cubic centimeters and precipitate the calcium as oxalate with ammonium oxalate in the usual way, as described in Art. **12**, taking all the precautions mentioned in the analysis of magnesia lime, Art. **13.** Calculate the results as described in Art. **12.**

Determination of Magnesium Oxide.—This is determined in the filtrate from the calcium-oxide determination by precipitation with ammonium oxalate in the usual manner (see Art. **12**, "Determination of Magnesium Oxide"). The weight of the magnesium pyrophosphate $Mg_2P_2O_7$ multiplied by the factor .36036 and by 100 gives the percentage of MgO in the agalite.

The following is an analysis of agalite:

Water (loss on ignition)	2.67%
Silica SiO_2	61.82%
$Fe_2O_3 + Al_2O_3$	1.59%
Lime CaO	3.65%
Magnesia MgO	29.98%
Manganese	trace
Undetermined	.29%
Total	100.00%

20. Analysis of Clay.—Clay is chemically a silicate of aluminum. Its value depends on the percentage of silicate of aluminum present and on the absence of iron oxide and grit. In analyzing clay, the following determinations are usually made as given.

Determination of Moisture.—Weigh out 8 grams of the clay in ground watch glasses. Dry in an air bath at 100° C. to constant weight. The loss of weight multiplied by 100 and divided by the weight of the sample taken gives the percentage of moisture in the sample.

Determination of Combined Water.—Weigh out 2 grams of clay in a platinum crucible and ignite at a red heat to constant weight. The loss of weight multiplied by 100 and divided by 2 gives the total percentage of water in the clay. Subtracting the percentage of moisture from this gives the percentage of combined water in the clay.

Determination of Silica.—Take 1 gram of the sample, heat in a platinum crucible at a low temperature for a few minutes, and then mix with 10 grams of a mixture of 1 part of sodium carbonate and 1 part of potassium carbonate. Fuse and proceed as directed for the determination of silica in Art. **19.** The weight of the silica multiplied by 100 gives its percentage in the clay. Collect the filtrate from the silica in a 200-cubic-centimeter flask and fill to the mark.

Determination of Sesquioxides of Iron and Aluminum.—Take 100 cubic centimeters of the filtrate from the silica determination (equivalent to .5 gram of clay), dilute to 200 cubic centimeters, and precipitate with NH_4OH in the usual manner. Proceed as in the same determination in Art. **12.** If a separation of the oxides is desired, use the methods given in the analysis of alum in Art. **18.** Calculate the results in the usual manner.

Determination of Calcium and Magnesium Oxides.—The filtrate from the iron- and aluminum-oxide determinations should be tested for CaO and MgO, and if present, they should be determined as directed in Art. **12.** The weights of the oxides found multiplied by 100 and divided by .5 gives the percentages of these oxides in the clay.

The following is an analysis of a good clay:

Moisture	.55%
Combined water	12.32%
Silica SiO_2	46.32%
Al_2O_3	40.25%
Fe_2O_3	.31%
CaO and MgO	trace
Undetermined	.25%
Total	100.00%

21. Analysis of Ocher.—Ochers are colored clays, their color being due to the presence of sesquioxide of iron Fe_2O_3. The greater the amount of iron, the deeper the color. The value of an ocher depends not only on its coloring power, but also on the absence of gritty substances, which tend to make the paper harsh and also injure the calenders. The following determinations and tests are usually made.

Grit Test.—The presence of *grit* is determined by means of a flotation test, which will, if carried out carefully, give the approximate amount of grit present. This test serves well for the comparison of two ochers.

Weigh out 5 grams of the ocher, transfer to a large beaker, fill with water, and stir for about a minute. Allow to settle 5 minutes and decant the cloudy portion. Repeat this operation until the water becomes perfectly clear, after settling 5 minutes. Filter, wash on the filter paper, dry, ignite, and weigh. When comparing two samples, weigh out equal amounts and treat both in exactly the same manner, when the better one in this respect can readily be determined. An idea as to the grit present can be determined by grinding a little of the ocher between the teeth.

Determination of Strength of Color.—A good quick method for testing two ochers as to *strength of color* is to mix them with zinc oxide and linseed oil, spreading on a piece of glass and comparing the depth of color.

Take .1 gram of a good strong ocher and mix well on a glass plate with 1 gram of zinc white ZnO, using as little linseed oil as possible. After it is thoroughly mixed and of uniform color, spread some of the mixture on a strip of glass. When the glass is turned over, the color produced by the ocher on the zinc white can be plainly seen. Now weigh out .1 gram of the ocher to be compared with the standard and mix with 1 gram of zinc white, as before, on a clean glass plate. Place this mixture alongside of the standard mixture on the glass, where the stronger one can be readily selected. By this method, one can keep mixing with larger and larger amounts of zinc white and determine approximately the per cent. of difference between the ochers compared.

For example, if it requires 1.1 grams of zinc white to produce the same shade with one ocher as 1 gram will produce with the same amount of the other, the former is 10 per cent. stronger than the latter. Two ochers may also be approximately compared as to the presence of sesquioxide of iron by weighing out equal amounts in test tubes, adding equal volumes of strong hydrochloric acid, placing the tubes in a beaker of boiling water for some time (until all the oxide of iron is dissolved), and removing both at the same time. The ocher containing the greater amount of sesquioxide of iron will color the acid the deeper.

Determination of Silica and Insoluble Matter.—Weigh out 1 gram of ocher and heat for some time in strong hydrochloric acid in a 4-inch porcelain dish. Evaporate slowly to dryness and drive off the excess of acid. Proceed as directed in the determination of silica in lime in Art. **12.** Weigh the residue as SiO_2 and insoluble matter. This weight multiplied by 100 gives the percentage in the ocher.

If it is desired to determine SiO_2 alone, fuse the above residue in 8 grams of a mixture of sodium and potassium carbonates in equal proportions. Proceed exactly as directed for the fusion of lime for silica in Art. **12.** Weigh as SiO_2, which weight multiplied by 100 gives its percentage in the ocher.

Determination of Sesquioxides of Iron and Aluminum.—These are determined in the filtrate from the silica and insoluble matter in the usual manner, as directed for the same determination in lime in Art. **12**.

Determination of Calcium and Magnesium Oxides.—If a qualitative test shows the presence of much of these oxides, they should be determined in the filtrate from the iron and aluminum-oxide determination, as directed in the analysis of lime in Art. **12** and the percentages calculated in the usual manner.

Determination of Moisture.—Weigh out between watch glasses 10 grams of ocher and dry to constant weight at 100° C. The loss of weight is due to moisture. This loss multiplied by 10 gives the percentage of moisture in the ocher.

Determination of Combined Water.—Weigh out 2 grams of ocher in a platinum crucible and ignite to constant weight at a low, red heat. The loss of weight multiplied by 50 will give the total percentage of water. Subtract the percentage of moisture found, and the difference will be the percentage of combined water.

The following is an analysis of a good ocher.

Moisture	.78%
Combined water	8.47%
Fe_2O_3	20.05%
Al_2O_3	7.11%
Silica SiO_2	62.62%
Lime CaO	trace
Undetermined	.97%
Total	100.00%

22. Analysis of Ultramarine.—Ultramarine is an artificial compound consisting of silica, alumina, sulphur, and soda. It is readily attacked by mineral acids, which destroy its color and liberate hydrogen sulphide. Its color varies from reddish to greenish blue. Ultramarine poor in silica is acted on by alum solutions, which destroy the color, while one rich in silica withstands this action. It is therefore advisable to use ultramarine rich in silica in paper manufacture. The following are the usual tests and determinations made in ultramarine:

Color Test.—Weigh out 2 grams of alum and dissolve in water in a 100-cubic-centimeter flask. Dilute to the mark and take two 50-cubic-centimeter samples of this alum solution in 100-cubic-centimeter flasks. Mix .2 gram of a good ultramarine in water in a 100-cubic-centimeter flask and fill to the mark. In another 100-cubic-centimeter flask mix up .2 gram of the sample to be tested and fill to the mark. After shaking well, take 10 cubic centimeters of each of the ultramarine mixtures and add to the above-mentioned alum solutions in the flasks. Allow them to stand for some time, with frequent shaking, and note the difference in color. The injurious effect of the alum can at once be told.

Determination of Moisture.—Weigh out between watch glasses 2 grams of the sample and dry to constant weight at 100° C., cooling in a desiccator. The loss in weight multiplied by 50 gives the percentage of moisture.

Determination of Silica.—Weigh out 1.5 grams of ultramarine, transfer to a porcelain dish, and treat with dilute hydrochloric acid; heat for some time; finally remove the cover glass to one side and evaporate to dryness. Proceed as directed for the determination of silica in lime in Art. **12.** Weigh as SiO_2 and earthy residue. This weight multiplied by 100 and divided by 1.5 gives the percentage of silica and earthy residue in the sample.

Determination of Sesquioxide of Aluminum and Iron. Take 100 cubic centimeters of the filtrate from the silica determination that has been made up to 300 cubic centimeters in a graduated flask and precipitate with ammonium hydrate in the usual way, proceeding as directed in the same determination in lime in Art. **12.** The weight of the ignited residue multiplied by 200 gives the percentage of Al_2O_3 and Fe_2O_3 in the sample.

Determination of Calcium Oxide.—If a qualitative test of the filtrate from the $Al_2O_3 + Fe_2O_3$ determination shows the presence of CaO, precipitate as oxalate, as directed for the same determination in lime in Art. **12.** Calculate in the usual manner.

Determination of Sulphuric Acid.—Take 100 cubic centimeters of the filtrate from the silica determination, dilute to 200 cubic centimeters, bring to a boil, and precipitate with barium chloride, as in the determination of H_2SO_4 in lime in Art. **12.** The weight of the $BaSO_4$ multiplied by .34335 × 200 gives the percentage of SO_3 in the sample.

Determination of Sodium Oxide.—Take the remaining 100 cubic centimeters of filtrate from the silica determination, precipitate, and wash the $Al_2O_3 + Fe_2O_3$ as before (this precipitate may be used as a check on the $Al_2O_3 + Fe_2O_3$ determination). Acidify this filtrate with H_2SO_4, evaporate to a small bulk, and transfer to a weighed platinum dish. Evaporate very cautiously on a water bath and finally over a low flame. At this point, care must be taken to avoid loss by sputtering. Heat cautiously until fumes of H_2SO_4 are no longer given off and then raise the temperature to red heat. Cool in a desiccator and weigh as Na_2SO_4. This weight will include $CaSO_4$ and $MgSO_4$, if calcium and magnesium are present. In the latter event, the amount of these elements present must be calculated to sulphates and the weight of these sulphates subtracted from the weight of the mixed sulphates to get the weight of pure Na_2SO_4. The weight of Na_2SO_4 multiplied by .43694 × 200 gives the percentage of Na_2O in the sample.

The extra precipitation of the $Al_2O_3 + Fe_2O_3$ may be avoided by running the filtrate from the original $Al_2O_3 + Fe_2O_3$ determination into a 200-cubic-centimeter flask, and using 100 cubic centimeters for the Na_2O and the other 100 cubic centimeters for the CaO determination. This, however, gives only a .25-gram sample to work on, and better results can be obtained by using a new sample.

Determination of Sulphur.—Weigh out 1 gram of the sample and mix in a platinum crucible with 2 grams of magnesium oxide and 1 gram of sodium carbonate, saving a little of the magnesium-oxide and sodium-carbonate mixture to cover over the top of the charge. Heat at a low, red heat for about 20 minutes and then stir with a platinum rod. Keep at this temperature, stirring from time to time, until the blue color disappears, which will take about 2 hours. Cool and wash into a small beaker with hot water, using a rubber-tipped glass rod to remove the last traces from the crucible. Add 10 cubic centimeters of bromine water, boil for about 1 minute, and filter. Wash well with hot water, add 10 cubic centimeters of hydrochloric acid, and boil until all the bromine is driven off. Precipitate with barium chloride, as in the determination of sulphuric acid in soda ash in Art. **10.** The weight of $BaSO_4$ multiplied by .13734 × 100 gives the percentage of sulphur in the sample.

The following is an analysis of ultramarine:

Silica and earthy residues	42.90%
Sulphur	11.78%
$Al_2O_3 + Fe_2O_3$	23.63%
Lime CaO	.16%
Soda $NaOH$	19.44%
Sulphuric acid	2.00%
Undetermined	.09%
Total	100.00%

23. Analysis of Rosin.—Rosin is the residue from turpentine distillation. It consists principally of the anhydride of abietic acid, the latter being $C_{18}H_{27}CO_2H$. It is readily soluble in alcohol and also in alkali solutions. The principal test of a rosin is for saponifiable rosin.

Test for Saponifiable Rosin.—Weigh out 2 grams of rosin, dissolve in 30 cubic centimeters of absolute alcohol, and pass a current of dry HCl gas through the solution until the gas is seen to escape. This will take about ¾ hour. Allow the flask to stand about 1 hour, to insure complete etherification. The resinic acids will separate on the top as an oily layer. Dilute the contents of the flask with five times its volume of distilled water and boil until the aqueous solution becomes clear. Transfer the contents of the flask to a separating funnel and rinse the flask with ether. Shake vigorously, allow to separate, run the acid layer off, and wash until the last trace of HCl is removed. Rinse the funnel with 50 cubic centimeters of alcohol into a beaker and titrate with normal sodium hydrate, using phenol phthalein as an indicator. The resinic acids combine at once with the alkali. Adopting 346 as the combining equivalent for rosin, the number of cubic centimeters of normal sodium hydrate used multiplied by .346 gives the amount of saponifiable rosin in the sample taken.

24. Analysis of Rosin Size.—When rosin and soda are boiled together, they combine to form resinate of soda, or rosin soap. The abietic acid and sodium carbonate are supposed to react according to the following equation:

$$2C_{16}H_{27}CO_2H + Na_2CO_3 = 2C_{16}H_{27}CO_2Na + CO_2 + H_2O$$

Rosin size prepared by different processes is analyzed by the same methods.

Determination of Water.—Weigh out about 20 grams of the thin size in a small beaker and dry at 100° C. to constant weight. This will take considerable time. The loss of weight multiplied by 100 and divided by the weight of the sample taken gives the percentage of water in the size.

Determination of Free Soda.—Weigh out about 20 grams of the thin size in a small beaker and dissolve in hot, strong alcohol. Filter, wash with the same alcohol, and heat the filtrate in the steam bath for a few minutes to drive off the alcohol. Take up the soda in hot water and titrate with standard acid, using methyl orange as an indicator. The number of cubic centimeters of acid used multiplied by the soda factor for each cubic centimeter, and this product multiplied by 100 and divided by the weight of sample taken, gives the percentage of free soda in the size.

Determination of Free Rosin.—Dissolve 20 grams as previously explained, and free from alcohol. Take up the residue with about 100 cubic centimeters of distilled water and transfer to a separating funnel. Add about 50 cubic centimeters of ether, agitate gently, and allow to separate. Draw the lower solution off as closely as possible, wash the ether solution several times with water, drawing the washings off into the original aqueous solution, and finally run the ether solution into a platinum dish that has previously been dried and weighed. Evaporate the ether, heat for a short time in the steam bath to drive off the moisture, and weigh. This weight, less the weight of the dish, gives the weight of rosin, which, multiplied by 100 and divided by the weight of the sample taken, gives the percentage of free rosin in the size.

Determination of Combined Rosin.—Return the aqueous solution from the previous determination to the separating funnel, add an excess of dilute sulphuric acid, which separates the combined rosin, shake with ether, and allow to separate. Draw off the lower layer, wash as before, and transfer to a weighed platinum dish. Proceed, and calculate the results exactly as in the previous determination.

Determination of Combined Soda.—The percentage of combined soda is usually estimated by difference, but it may be checked by taking the sample in which the moisture was determined, taking up with about 75 or 100 cubic centimeters of hot water, cooling, and titrating with a standard acid, using methyl orange as an indicator. This gives the total soda in the sample. The combined soda is found by subtracting the amount of free soda determined previously. Calculate the percentage in the usual manner.

The following are analyses of various sizes:

	Brown Size	Dry Basis
Water	86.350%	
Free soda	.012%	.088%
Free rosin	2.560%	18.754%
Combined rosin	9.180%	67.253%
Combined soda	1.830%	13.407%
Undetermined	.068%	.498%
Total	100.000%	100.000%

	Brown Size (With Small Amount of Free Rosin)	Dry Basis
Water	86.370%	
Free soda	.800%	5.87%
Free rosin	.296%	2.17%
Combined rosin	10.920%	80.12%
Combined soda	1.610%	11.81%
Undetermined	.004%	.03%
Total	100.000%	100.00%

	White Size	Dry Basis
Water	97.8000%	
Free soda	trace	trace
Free rosin	.7145%	32.48%
Combined rosin	1.2681%	57.64%
Combined soda	.2149%	9.77%
Undetermined	.0025%	.11%
Total	100.0000%	100.00%

25. Testing of Glue or Animal Size.—This substance is made by heating the clippings of bones, horns, etc. with water. The best grades of glue for paper-mill use have been thoroughly washed, and are ready for mixing up for sizing purposes without any previous purification.

Glue is insoluble in cold water, but will absorb four or five times its weight of water and swell up. It is readily soluble in hot water, the solution gelatinizing on cooling.

The following tests may be made to determine the quality of a glue for paper makers' use:

Determination of Power of Absorption. — Weigh out 10 grams of the sample into a beaker and fill with cold water, taking care that all of the glue is covered by the water. Do not allow the temperature to rise above 12° C. for twenty-four hours. At the end of that time pour off the excess of water and weigh again. The increase of weight will indicate the amount of water absorbed. The absorptive power is expressed as the number of times its weight of water absorbed.

Determination of Moisture and Ash. — The moisture is determined by drying a sample of the glue to constant weight at 100° C., the loss of weight being considered as moisture. For the determination of the ash ignite the same sample and weigh the ash.

Comparison of the Value of Glues.—A good, practical test that serves as a comparison of two glues as to money value is made as follows: Weigh out 10 grams of the higher priced glue in a small beaker, then weigh out an amount of the lower priced glue, which will be equivalent in price to 10 grams of the former. Add 100 cubic centimeters of water to each at the same time, heat gently, keeping both at the same temperature, and stirring in the same manner, until all the glue is dissolved. Remove and allow to cool and gelatinize. Compare the strength of the two mixes. If neither gelatinizes, it will be necessary to take larger samples. A good glue for paper makers' use should gelatinize at 75° F. when mixed in the proportion of 10 grams of glue to 100 cubic centimeters of water.

ILLUSTRATION.—Two glues marked *A* and *H*, and costing 12¼ cents and 12 cents, respectively, were compared. Treated as above, it required 11.5 grams of *H* to gelatinize from the same amount of water as 10 grams of *A*. Therefore, if *H* be worth 12 cents per pound, *A* is worth 13.8 cents, and since *A* sells at 12¼ cents per pound, other things being equal, it is by far the cheaper glue.

26. Analysis of Salt.—Common salt, or sodium chloride, is used in connection with the paper mill for the preparation of a bleach solution by electrolysis. The crude salt is used, but it should be as free as possible from calcium chloride. The following determinations are usually made:

Determination of Moisture.—Weigh out 5 grams of salt in a covered platinum crucible. Heat gently at first over a low flame, gradually raising the temperature to a low red heat. Keep at this temperature for a few minutes, cool in a desiccator and weigh. The loss in weight multiplied by 20 gives the percentage of moisture in the salt. During the heating, great care should be taken to avoid loss by sputtering.

Determination of Insoluble Matter.—Weigh out 5.85 grams of salt. Dissolve in hot water and filter into a 500-cubic-centimeter flask. Wash well with hot water, dry, ignite, and weigh. This weight multiplied by 100 and divided by 5.85 gives the percentage of insoluble matter in the salt.

Determination of Chlorine. —Cool the filtrate from the previous determination and fill to the mark. Take 25 cubic centimeters of this in a beaker by means of a pipette, dilute to about 50 cubic centimeters, add a little potassium-chromate indicator, and titrate with $\frac{n}{10}$ silver nitrate solution. When the solution changes from yellow to faint pink, take the reading. The number of cubic centimeters required multiplied by 2 gives the percentage of sodium chloride in the sample. This percentage multiplied by .6239 gives the percentage of chlorine.

Determination of Calcium Oxide.—Dissolve 5 grams of salt in water and a little hydrochloric acid, if necessary. Filter off the insoluble matter and make alkaline with ammonia. Precipitate the calcium as oxalate in the usual manner, as described in Art. **12,** in the determination of calcium oxide.

If there is no sulphuric acid in the sample, the calcium oxide found is calculated to the chloride, and its equivalent of $NaCl$ subtracted from the $NaCl$ found.

Determination of Sulphuric Acid. —If a qualitative test shows the presence of sulphuric acid, it should be determined in the usual manner by precipitating with barium chloride, as has been described. The weight of $BaSO_4$ multiplied by $100 \times .34335$ and divided by the weight of the sample taken gives the percentage of SO_3 in the salt.

The following is an analysis of salt used:

Moisture	.480%
Insoluble matter	.038%
Calcium chloride $CaCl_2$	1.100%
Sodium chloride $NaCl$	97.690%
Calcium sulphate $CaSO_4$	trace
Undetermined	.692%
Total	100.000%

27. Analysis of Sulphur or Brimstone.—Sulphur is used in the manufacture of bisulphite liquor. For every 100 kilograms of sulphur there should be a yield of 190 kilograms of sulphur dioxide. A good sulphur should contain less than 1 per cent. of foreign matter.

Determination of Moisture.—An average sample of from 50 to 75 grams of sulphur should be taken and dried, without grinding, at 100° C. for 12 hours. If the sample is first ground, some of the moisture will be lost by evaporation. The results are calculated in the usual manner.

Determination of Ash.—Burn 10 grams of sulphur in a weighed porcelain dish, weigh, and calculate the percentage of ash.

Determination of Sulphur.—FIRST METHOD.—Weigh out 50 grams of the finely ground sulphur and digest in 200 cubic centimeters of carbon bisulphide in a stoppered bottle at the ordinary temperature. Take the specific gravity of the liquid. Reduce this to specific gravity at 15° C. by means of the following formula:

$$D = g + .0014\ (t - 15),$$

where $D =$ specific gravity at 15° C.
$g =$ specific gravity at actual temperature;
$t =$ temperature (deg. C.) of solution.

This formula will hold good up to 25° C. According to a carefully computed table, which is given below, the percentage of sulphur in the sample of brimstone is given for each specific gravity of the above solution.

TABLE II

PERCENTAGE OF SULPHUR IN THE SAMPLE FOR EACH SPECIFIC GRAVITY, PROVIDED THE FOREGOING PROPORTIONS ARE USED

Sp. Gr.	% S	Sp. Gr.	% S	Sp. Gr.	% S	Sp. Gr.	% S	Sp. Gr.	% S	Sp. Gr.	% S
1.282	10.4	1.297	25.2	1.312	39.6	1.327	54.0	1.342	68.4	1.357	84.0
1.283	11.6	1.298	26.0	1.313	40.8	1.328	55.2	1.343	69.6	1.358	84.8
1.284	12.4	1.299	26.8	1.314	41.6	1.329	56.0	1.344	70.4	1.359	86.0
1.285	13.6	1.300	28.0	1.315	42.4	1.330	56.8	1.345	71.6	1.360	87.2
1.286	14.4	1.301	28.8	1.316	43.6	1.331	58.0	1.346	72.4	1.361	88.4
1.287	15.6	1.302	30.0	1.317	44.4	1.332	58.8	1.347	73.6	1.362	89.2
1.288	16.4	1.303	31.2	1.318	45.2	1.333	60.0	1.348	74.4	1.363	90.8
1.289	17.6	1.304	32.0	1.319	46.4	1.334	60.8	1.349	75.6	1.364	92.0
1.290	18.4	1.305	32.8	1.320	47.2	1.335	61.6	1.350	76.0	1.365	92.8
1.291	19.2	1.306	34.0	1.321	48.4	1.336	62.4	1.351	77.2	1.366	94.4
1.292	20.0	1.307	34.8	1.322	49.2	1.337	63.6	1.352	78.4	1.367	96.0
1.293	21.2	1.308	35.6	1.323	50.4	1.338	64.4	1.353	79.6	1.368	97.2
1.294	22.4	1.309	36.8	1.324	51.2	1.339	65.6	1.354	80.4	1.369	99.2
1.295	23.2	1.310	37.6	1.325	52.4	1.340	66.4	1.355	81.6		
1.296	24.0	1.311	38.8	1.326	53.2	1.341	67.6	1.356	82.4		

Second Method.—Weigh out .5 gram of the sample into a porcelain dish of 250 cubic centimeters capacity. Heat to 30° C. and add 6 cubic centimeters of bromine. After keeping at this temperature for about 10 minutes, add 15 cubic centimeters of concentrated nitric acid heated to 30° C. A violent action takes place with the formation of sulphuric and hydrobromic acids, the remaining bromine distilling off. Heat with caution at first and then bring to a boil in order to drive off the nitric acid. Add a small amount of a solution of $NaCl$ to avoid loss of sulphuric acid when evaporating and evaporate to small bulk. Add hydrochloric acid and evaporate to small bulk again and repeat this three or four times, finally evaporating to dryness. Heat gently to drive off hydrochloric acid and take up with 5 cubic centimeters of half-strength hydrochloric acid and about 100 cubic centimeters of water. Filter into a 500-cubic-centimeter flask and wash well with hot water. The residue can be ignited and weighed for the determination of insoluble matter.

Dilute filtrate to the mark, take 100 cubic centimeters and precipitate the sulphur as $BaSO_4$ with barium chloride in the usual manner, filter, dry, ignite, and weigh as $BaSO_4$ and calculate to sulphur by multiplying by .13734. The weight of the sulphur thus found multiplied by 1,000 gives its percentage in the sample.

The following is an analysis of sulphur:

Moisture	.24%	
Foreign matter	.63%	
Sulphur	99.13%	
Total	100.00%	Ash, .27%

28. Analysis of Bisulphite Liquor.—This liquor, which is used in the manufacture of "sulphite pulp," consists principally of calcium bisulphite $CaH_2S_2O_6$, magnesium bisulphite $MgH_2S_2O_6$, and calcium sulphate $CaSO_4$, together with free sulphur dioxide SO_2.

The ordinary tests made to control the working of the liquor are for sulphur dioxide SO_2, by titrating with

$\frac{n}{10}$ iodine solution, using starch as an indicator; and for acidity, by titrating with $\frac{n}{10}$ sodium-hydrate solution, using phenol phthalein as an indicator. For each of these tests, 2 cubic centimeters of liquor should be used.

A complete analysis of the liquor is often required and can be made according to the following methods.

Determination of Silica.—Take 10 cubic centimeters of the liquor in a porcelain dish and weigh; add 5 cubic centimeters of hydrochloric acid and evaporate to dryness. Drive off the excess of acid by gently heating, and take up in 5 cubic centimeters of dilute hydrochloric acid. Dilute to about 100 cubic centimeters, boil, filter, wash well with hot water, dry, ignite, and weigh as SiO_2.

The specific gravity of the liquor should be taken, which, multiplied by 10, gives the weight in grams of the liquor taken for the test. The weight of silica found, multiplied by 100 and divided by the weight of liquor taken, gives the percentage of silica.

Determination of Sesquioxides of Iron and Aluminum. Treat the filtrate from the silica determination with NH_4OH to precipitate the hydrates of iron and aluminum. Proceed as in Art. **12.** The weight of the oxides multiplied by 100 and divided by the weight of liquor taken, as found in the silica determination, gives the percentage of these oxides in the liquor.

Determination of Calcium Oxide.—Concentrate the filtrate from the preceding determination to about 200 cubic centimeters. Heat to boiling and precipitate the calcium as oxalate with ammonium oxalate, as in Art. **12.** Calculate the percentage of CaO in the usual manner.

Determination of Magnesium Oxide.—Evaporate the filtrate from the calcium-oxide determination and proceed as directed in Art. **12.** The weight of the magnesium pyrophosphate multiplied by $100 \times .36036$ and divided by the weight of liquor taken gives the percentage of MgO in the sample.

Determination of Sodium Oxide.—If care is taken, the filtrate from the magnesium-oxide determination (provided ammonium phosphate were used to precipitate the magnesia) can be evaporated to a small bulk, transferred to a platinum dish, evaporated to dryness on the water bath, ignited carefully after converting the sodium to sulphate by treating with a few drops of sulphuric acid. The excess of acid is driven off and the weight found and the percentage determined as given below.

As there is great danger of loss from sputtering, it is better to take a new sample of 25 cubic centimeters of liquor, precipitate the heavy bases with ammonium carbonate, filter, and wash, and evaporate the filtrate in a platinum dish, after treating with a little dilute sulphuric acid. Ignite until all sulphuric acid is driven off, cool, and weigh as Na_2SO_4. The weight of the Na_2SO_4 multiplied by .43694 × 100 and divided by the weight of the sample taken gives the percentage of sodium oxide.

Determination of Sulphuric Acid.—Treat 10 cubic centimeters of the liquor in a porcelain dish with 5 cubic centimeters of hydrochloric acid and evaporate nearly to dryness to drive off the sulphur dioxide; dilute and precipitate with $BaCl_2$. If it should come to dryness, take up with a little hydrochloric acid, dilute, and precipitate with barium chloride, as directed in Art. **12.** The weight of $BaSO_4$ multiplied by 100 × .34335 and divided by the weight of the sample taken gives the percentage of SO_3 in the liquor.

Determination of Sulphur Dioxide.—Oxidize 10 cubic centimeters of the liquor with 10 cubic centimeters of bromine water, pouring the liquor into the bromine water. Heat, dilute, and boil to drive off the excess of bromine. Precipitate with barium chloride, as in Art. **12.** Calculate the SO_3 as in the previous analysis and subtract the amount found there from the amount just found. The difference will be the SO_3 from the oxidation of the SO_2. This weight multiplied by 100 × 18001 and divided by the weight of the sample taken gives the percentage of SO_2 in the liquor.

In calculating the results of the liquor analyses, the sulphuric acid is combined with lime to form calcium sulphate $CaSO_4$. If there be less lime than is necessary to saturate the SO_3, the balance of the SO_3 is calculated to magnesium sulphate $MgSO_4$.

Any excess of lime is calculated to calcium bisulphite $CaH_2S_2O_6$, and if there be more SO_2 than is necessary to combine with all the lime, the magnesium oxide is calculated first to the monosulphite $MgSO_3$, and if there be an excess of SO_2, to the bisulphite $MgH_2S_2O_6$. Any further excess is reported as free SO_2.

The following is an analysis of a good bisulphite liquor:

Silica SiO_2	.07%
$Fe_2O_3 + Al_2O_3$	.02%
Bisulphite of lime $CaH_2S_2O_6$	2.76%
Bisulphite of magnesia $MgH_2S_2O_6$	3.19%
Calcium sulphate $CaSO_4$	.16%
Free sulphur dioxide SO_2	.14%
Water and undetermined	93.66%
Total	100.00%

29. Analysis of Caustic Liquor.—This liquor is used for digesting the wood in the soda process. Its strength varies in different mills from 11° to 12° Baumé at 60° F. In order to get good results, the liquor should be from 93.5 to 94 per cent. caustic; that is, this percentage of the soda present should be caustic. This test is practically the only one made.

Determination of Causticity.—Make up a weak solution of sulphuric acid (about 6 cubic centimeters of strong acid to 1 liter of water is a convenient strength). Take about 5 cubic centimeters of the caustic liquor (the amount should be varied according to the strength, but never using enough to require more than 50 cubic centimeters of the acid), dilute in a small beaker to about 50 cubic centimeters, add a few drops of phenol phthalein. Titrate with the dilute sulphuric acid until the pink color just leaves and take a reading.

Now add a drop of methyl-orange solution, titrate cautiously until a slight pink tint is reached, and take a second reading.

Subtract the first reading from the second, multiply the difference by 2, and subtract from the second reading. This remainder divided by the second reading and the result multiplied by 100 gives the percentage of causticity.

ILLUSTRATION.—The first reading when the pink color disappeared was 42.6 cubic centimeters. After adding methyl orange and titrating again until pink, the reading was 43.9 cubic centimeters. Following out the calculation as explained,

$$\begin{aligned} 43.9 - 42.6 &= 1.3 \\ 1.3 \times 2 &= 2.6 \\ 43.9 - 2.6 &= 41.3 \\ 41.3 \div 43.9 &= .9407 \\ .9407 \times 100 &= 94.07 \text{ per cent. caustic.} \end{aligned}$$

Another method that gives very accurate results is as follows: Measure out 25 cubic centimeters of the caustic liquor and titrate with a normal acid, using methyl orange as an indicator. This gives the total alkali. Take 100 cubic centimeters of the same liquor and precipitate the carbonate and sulphate with barium chloride in slight excess. Dilute to the mark in a 200-cubic-centimeter flask, shake well, and allow to settle. Take 50 cubic centimeters of this clear solution and titrate with the normal acid, using phenol phthalein as an indicator. This will give the amount of $NaOH$ in 25 cubic centimeters of the caustic liquor. The number of cubic centimeters of acid used in the second titration divided by the number of cubic centimeters used in the first titration and multiplied by 100 gives the percentage of causticity.

30. Color Value of Pigments.—The price of a pigment does not always indicate its value. This is determined by its coloring power. A chemical analysis will determine the value of a pigment, but there are simple methods of comparing colors and dyes, which can be operated by one not having a knowledge of chemistry. Under the analysis of ochers in Art. **21**, a method was given for comparing colors, by mixing the ocher with zinc white and linseed oil. A similar method to this may be used for comparing all colors and dyes.

Comparison of Pigments.—To compare two pigments, weigh out 1 gram of the higher-priced one and mix with 10 grams of a good, dry china clay. Now weigh out as many grams of the lower-priced pigment as can be bought for the price of 1 gram of the higher-priced one and mix with 10 grams of the same clay. Mix separately in a mortar with water to a thin, pasty mass. Spread each upon a strip of glass side by side and dry in a steam bath. The colors can now be compared, and the one having the higher color value selected. This test may be carried still farther by weighing out a larger amount of clay and adding to the one giving the strongest tint in the above test until it matches the other. By noting the difference in the amount of clay used, the difference in value of the two pigments can be determined.

Comparison of Soluble Colors.—A method used to a considerable extent in paper mills for comparing soluble colors consists in weighing out different amounts of the substances in proportion to their prices, dissolving in 1 liter of water, taking 20 cubic centimeters of each solution, diluting to a light tint, and matching the tints. By noting the amount of water required in each cylinder, the tinctorial values may be calculated.

ILLUSTRATION.—Two samples of soluble blue, costing 15 cents and 25 cents, respectively, were compared as follows: Weighed out 1.5 gram of the first and .9 gram of the second, these weights representing equal values, dissolved in water, and made each solution up to 1 liter. Took 20 cubic centimeters of each solution, diluted in the cylinders to a light tint, and matched the tints. It required 120 cubic centimeters of water for the first and 160 cubic centimeters of water for the second, making their tinctorial value as 140 and 180 per unit of cost.

From the above it is found that 1 pound at 15 cents is required to color 140 pounds of pulp to a given shade, and .6 pound at 25 cents is required to color 180 pounds of pulp to the same shade; or, roughly, .7 pound at 15 cents and .33 pound at 25 cents are required to color 100 pounds of pulp to the same shade. Therefore, it will cost 10.5 cents to color 100 pounds of pulp using the lower-priced blue, while it will cost but 8.25 cents to color 100 pounds of pulp to the same shade using the higher-priced blue.

31. Analysis of Wood Pulp.—The analysis of soda and sulphite pulps are taken up under separate heads.

Determination of Cellulose in Wood.—Weigh out 5 grams of the finely divided substance and boil four or five times in water, using 100 cubic centimeters each time. Pour off the water and dry at 100° C. Now exhaust with a mixture of equal parts, by measure, of benzine and strong alcohol, to remove the rosin, fat, wax, etc. Dry the residue and boil several times in water, to every 100 cubic centimeters of which 1 cubic centimeter of strong ammonia has been added. This treatment removes the pectous substances and coloring matter. Treat the residue in a closed bottle with 250 cubic centimeters of water and 20 cubic centimeters of bromine water, containing 4 cubic centimeters of bromine to the liter. When the yellow color of the liquid disappears, add more bromine water and repeat until the yellow color remains and bromine can be detected after standing 12 hours. Filter off the liquid, wash the residue with water, and heat to boiling with 1 liter of water containing 5 cubic centimeters of strong ammonia. If the liquid and tissue are colored brown, filter off the undissolved matter, wash, and treat again with bromine water, as above. When the action seems complete, heat the residue as before with ammonia water, and if the brownish tint is again imparted to the liquid, repeat the above operation. The cellulose is thus obtained in a pure, white state. Wash with boiling water, then with hot alcohol. Dry at 100° C. and weigh. The percentage of cellulose is calculated in the usual manner.

The following table gives the percentages of cellulose in representative woods as determined by the above method:

TABLE III

Wood	Percentage of Cellulose	Wood	Percentage of Cellulose
Alder	54.61	Oak	39.50
Beech	45.49	Pine	57.00
Birch	55.52	Poplar	62.80
Chestnut	52.64	Scotch pine	53.27
Ebony	30.00	Spruce	53.00
Fir	53.30	Willow	55.72
Linden	55.09		

32. Analysis of Soda Pulp.—This pulp is made by the soda process. Samples from the rolls and from the wet and pulp machine should be taken from time to time and tested.

In taking samples from the roll for shipment, cut through at least ten layers of the pulp and take two strips about 12 inches long and ½ inch wide. Tear these into small pieces and put into a weighed sample box, keeping the lid on as much as possible. A sample should be taken from every tenth roll.

Determination of Total Moisture.—Weigh the box containing the sample and empty upon a clean piece of paper in the drying oven. Dry at 100° C. for about 6 hours. Return the sample to the box, cool in a desiccator, and weigh again. Repeat until the weight is constant. The loss in weight multiplied by 100 and divided by the weight of the sample taken gives the percentage of moisture in the pulp.

The folds from the wet machine are tested in the same way.

Determination of Air-Dry Moisture.—**Air-dry pulp** is the term given to pulp that only contains moisture due to the atmosphere. This is rather indefinite, owing to the difference in humidity. It is determined by drying a sample until bone dry, weighing, and exposing to atmosphere at ordinary temperature for at least 24 hours. The increase in weight will indicate the amount of air-dry moisture.

Determination of Ash.—Weigh out about 5 grams of the pulp sample in a platinum crucible. Ignite until all carbonaceous matter is burned off. Cool in a desiccator and weigh. The weight of the ash multiplied by 100 and divided by the weight of the sample taken gives its percentage in the pulp. Soda pulp that has been run over the driers usually contains from 7 to 8 per cent. of moisture and about 1 per cent. of ash.

Pulp after leaving the last press roll and before passing over the driers contains about 65 per cent. of moisture.

33. Analysis of Sulphite Pulp.—This pulp is made by the sulphite process. It is shipped in folds from the wet machine, and contains from 60 to 70 per cent. of moisture.

Determination of Moisture.—Cut strips 10 inches long and ½ inch wide from folds taken at random from different parts of the lot sampled, taking care that a good average sample is selected. Tear these strips into small pieces and put into a sample box. Weigh the box and contents and transfer the sample to the drying oven. Dry at 100° C. for from 18 to 24 hours. Return to the box and weigh again. Subtract the weight of the box to get the weight of bone-dry fiber. Add 10 per cent. of this weight and call this the weight of the air-dry fiber.

The weight of the air-dry fiber subtracted from the weight of the sample taken gives the excess of moisture. This weight multiplied by 100 and divided by the weight of the sample taken gives the percentage of excess of moisture in the pulp.

The following is a convenient method for the determination of moisture, which gives results sufficiently accurate for all practical purposes. The results are expressed in percentage of air-dry fiber. This method is used when samples are taken from time to time from the wet machine to ascertain how the pulp is running and to figure out the percentage of fiber made daily.

Take 100 grams of the sample, weighing as quickly as possible, and transfer to the drying oven, after separating the different layers as much as possible. Dry from 18 to 24 hours and rapidly weigh again. The weight of the dried sample divided by 90 and multiplied by 100 gives the percentage of air-dry fiber.

Determination of Ash.—The ash is determined as for soda pulp in Art. **32.** For sulphite pulp it is usually from .6 to 1 per cent.

34. Paper Testing.—Samples of paper are tested in the same way as pulp for moisture and ash, taking about 50 grams for the determination of moisture and 3 or 4 grams for the ash.

Determination of Retention of Filler.—It is important to make frequent tests of the paper for ash in order to figure out the retention of the filler used.

ILLUSTRATION.—A beater contained 1,000 pounds of stock when furnished, and 200 pounds of agalite were used in furnishing it; 20 per cent. of the mix therefore was agalite. The paper made from this mix was tested for ash, and found to contain 14.83 per cent. Allowing 1 per cent. for ash other than filler gives 13.83 per cent. filler. 13.83 divided by 20 gives 69.15 per cent. retention.

Microscopic Fiber Test.—The various fibers in paper are detected by means of a microscopic examination, while at the same time there are several chemical tests that are useful in identification.

Prepare the sample to be examined under the microscope as follows: Cut small pieces of paper from different parts of the sample and boil in a 1-per-cent. solution of caustic soda. The fibers may now be separated by shaking in a bottle containing a few pieces of broken glass. The fibers are placed on a glass, covered with a drop of glycerine, and a cover glass.

Mechanical Wood Fiber.—Under the microscope, ***mechanical wood fiber*** is distinguished from ***chemical wood fiber*** by rarely having the fibers separated, they being generally bound together in bundles. The ends also are torn and jagged.

Linen Fiber.—Fig. 3 is an illustration of *linen fiber.* The ends are usually drawn out into numerous fibrils and fibers of a cylindrical form.

Esparto and Straw Fibers.—These are very much alike, and consist of serrated cells and fibro-vascular bundles. Straw may be distinguished from esparto by the presence of small, oval-shaped cells, and the absence of the fine hairs that always line the inner surface of the leaf of the esparto plant.

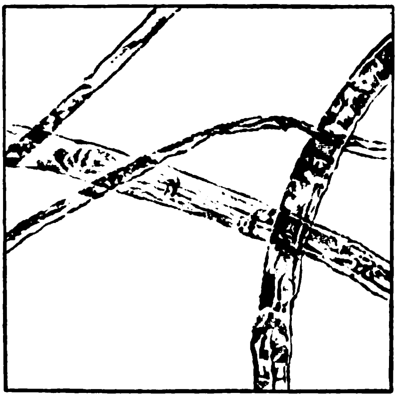

FIG. 3

Chemical Wood Fiber.—These fibers are flat and ribbon-like and have unbroken ends, very much like cotton, but are distinguished from it in that they are not twisted. Pine-wood fiber is detected by the presence of small pitted vessels.

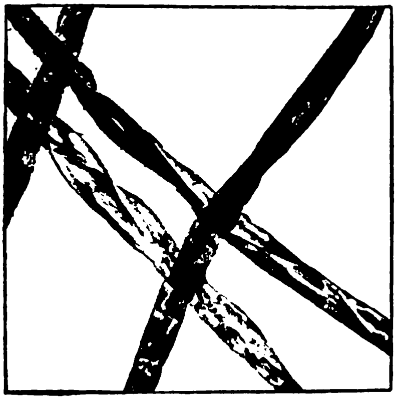

FIG. 4

Cotton fiber is shown in Fig. 4, poplar in Fig. 5, and spruce in Fig. 6.

Chemical Tests for Constituents of Paper. When a small quantity of paper that has been beaten up, separated, and moistened with iodine is examined under the glass, the fibers can be separated, owing to the fact that the ground wood and jute will be colored yellow; cotton hemp and linen, brown; and bleached straw, esparto, and chemical wood fiber will remain uncolored.

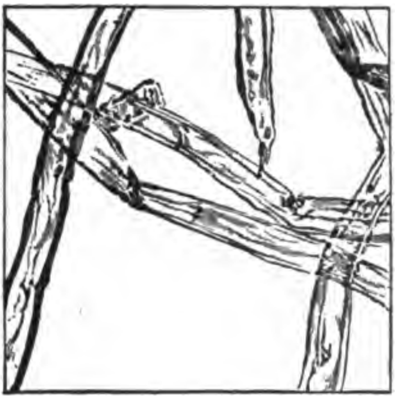

FIG. 5

Detection of Mechanial Wood Pulp.—Nitric acid gives a brown stain on paper containing mechanical wood pulp. Phloroglucinol gives a deep magenta in the presence of mechanical wood pulp. The latter test is the one mostly used for this purpose. Phloroglucinol is prepared by dissolving 2 grams of the reagent in 25 cubic centimeters of 95-per cent. alcohol and adding 5 cubic centimeters of concentrated hydrochloric acid. Apply a drop to the paper to be tested and allow to evaporate. In the presence of ground-wood pulp the deep magenta coloration will be developed.

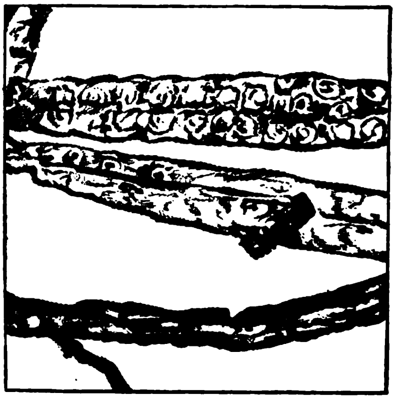

FIG. 6

The *percentage* of ground wood may be roughly estimated by comparing the depth of color produced by the phloroglucinol on the sample with the depth of color produced on several standard samples, in which the percentage of ground wood is known. This test can be applied only when the chemical fiber present has been perfectly reduced.

Detection of Sulphite Pulp.—This can be detected by means of a dilute solution of sodium-auric chloride, which imparts to the paper moistened with it a reddish-brown color when unbleached sulphite pulp is present, and a bluish color when bleached sulphite pulp is present.

Detection of Straw and Esparto.—These can be detected by boiling the paper for some time in a 1-per-cent. solution of aniline sulphate, which produces a red color in the presence of these substances.

Detection of Animal Size.—Heat a small fragment of paper in a test tube with distilled water, transfer the clear solution to another tube, cool, and add a solution of tannic acid. If any animal size be present, a milky, flocculent precipitate will be formed, the consistency of which will depend on the amount of animal size present.

A very delicate test for animal size is to soak a small strip of paper in a reagent prepared by dissolving a small amount of quicksilver in an equal weight of fuming nitric acid, cooling, and adding an equal volume of water. In the presence of animal size, the paper will develop a red color, the depth of which will depend on the amount of size present. This reagent will keep but about a month, and hence must be made up frequently.

Detection of Starch.—Starch may be detected in paper by adding a drop of very dilute solution of iodine, which, if starch is present, will develop a deep-blue color. This test is better carried out by boiling some of the paper in water for about 20 minutes, pouring off the water into another vessel, and adding the dilute iodine solution.

Detection of Rosin Size.—The presence of rosin size may be detected by heating some of the paper in absolute alcohol and pouring the alcoholic solution, after cooling, into five times its volume of distilled water. If a precipitate is formed or there is a cloudiness, the presence of rosin size is indicated.

By wetting the sheet in the mouth and holding it to the light, a well-sized paper will not be transparent, while if poorly sized, it will be transparent, the degree of transparency varying inversely with the amount of size used.

Detection of Chlorides.—Boil the paper in distilled water, filter, add a few drops of nitric acid, and then a few drops of silver-nitrate solution. A cloudiness or precipitate indicates the presence of chlorides.

Detection of Alum.—An excess of alum can be detected by boiling a quantity of the paper in a small amount of water, filtering, and testing the filtrate for aluminum by precipitating with ammonium hydrate and ammonium chloride.

The *nature* of the filler can be determined by an analysis of the ash.

Determination of Coloring Matters.—Sometimes coloring matters can be determined by an examination of the ash. When ultramarine has been used, the ash is blue.

35. Mechanical Tests of Paper.—There are several mechanical tests to which paper should be subjected in order to determine its quality.

Determination of the Direction in Which the Paper Came From the Machine.—This test is necessary to determine the strength of paper. Cut a disk from the center of the sheet of paper, float it upon water, and allow it to rest upon the palm of the hand, when it will curl up. The direction of the axis of the cylinder formed is the direction in which the paper came from the machine.

Determination of the Strength of Paper.—The strength of paper is determined roughly by tearing the sheet. Two papers can be compared in this respect by means of a testing machine, for which test strips are cut (taking care that they are cut in the same way, as to coming from machine and are of the same size). The strips are then subjected to tension in the testing machine, and the weight necessary to break them read off and compared. Two sheets can be roughly compared in this respect by cutting strips an inch in width from each sample, and suspending them from an iron bar. Weights are then cautiously attached to each until they break; the difference in weights required will give the comparative strength of sheets.

Detection of Dirt.—Dirt in paper can be detected by holding the sheet to a light.

MANUFACTURE OF SUGAR

(PART 1)

CANE SUGAR

INTRODUCTORY

1. Definition.—The term sugar was originally employed and intended to classify all substances having a sweet flavor, and thus came to be used almost indiscriminately for cane sugar, fruit sugar, lead acetate (so-called sugar of lead), and other compounds possessing this property. At present, in a general sense, it is reserved almost exclusively to denote *cane* and *beet sugar*, that is, the true crystallizable sugar or *sucrose*.

In chemistry, the word sugar is applied to a large class of organic bodies belonging to the group of carbohydrates. These carbohydrates may be subdivided into two groups: those that, disregarding the water of crystallization, have the composition $C_n(H_2O)_n$ and those that correspond to the formula $C_n(H_2O)_{n-1}$. The first-named group includes dextrose or glucose (which, when it occurs naturally, should be known as *grape sugar*, *starch sugar*, etc.), levulose, arabinose, lactose, and others. The last-named group embraces sucrose (cane and beet sugar), maltose, galactose, and others.

The sugars of the formula $C_n(H_2O)_{n-1}$, on treatment with dilute acids, undergo inversion, that is, they take up the elements of water and are converted to equal quantities of

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the sugars having the formula $C_n(H_2O)_n$, one of which is always glucose.

2. History of Sugar. — Etymologically, sugar would seem to be of Indian origin, the earliest forms of the word being *sarkara* in Sanskrit and *sakkara* in Prakrit. Thence it may be traced through all the Aryan languages, as *schakar* in Persian, *sukkar* in Arabic, *suicar* in Assyrian and Phenician, *saccharum* in Latin, *azucar* in Spanish and Portuguese, *zuchero* in Italian, *sucre* in French, *zucker* in German, etc.

The precise product indicated by these various names is not always clear, and probably is not identical in all cases. The cultivation of the genuine sugar cane (*Saccharum spp.*) appears to have been common in India and China in very remote times, but there is no documentary evidence on this point earlier than Herodotus. Frequent mention of the "sweet cane" occurs in the Scriptures, but the plant referred to is doubtful. An Indian reed yielding honey is alluded to by Strabo, and a similar statement concerning an Egyptian reed is made by Theophrastus; while Dioscorides actually gives the name *saccharum* to a kind of honey obtained from reeds in Arabia Felix and India; both he and Pliny accurately describe the product as being white and brittle and of a salt-like consistency. Later it seems to have been generally termed *Indian salt* among the Greeks and Romans, by whom it was obtained in small quantities at great expense from India, and used medicinally.

The introduction of cane sugar into the Mediterranean basin must have occurred at an early date; for it was found growing at Assouan, on the Nile, in 714, and was carried into Spain by the Moors in 766, while Sicily engaged in the culture about 1060 to 1090. During the religious wars of the Middle Ages, the "sweet honeyed reeds," called *zucra*, which abounded in the meadows about Tripoli, were consumed by the Crusaders; and it is evident that sugar making in that neighborhood was conducted in a wholesale and systematic manner. From Cyprus and Madeira, the industry extended, in 1500 to 1600, to most of the West Indies, where it was carried on by Spanish and British colonists; but there is strong evidence in favor of the supposition that several kinds of sugar cane are indigenous both to the West Indies and to almost the whole continent of South America.

3. From the extensive growth of sugar in the Western Tropics there ensued large importations of the raw article into Europe; the introduction of tea and coffee about the same time created a general and wide demand for what had hitherto been regarded as a medicine rather than a nutritive article of diet. Sugar refining appears to have been copied from the Arabs by the Venetians, and refineries were established in England and Germany in the 16th century, and in Holland soon after.

4. Up to this time, cane sugar was the only kind known in commerce. But, in 1747, Margraf, a German chemist, demonstrated the existence of about 6 per cent. of sugar in beet root; and in 1795, Achard manufactured beet sugar on his farm in Silesia, Germany, and presented loaves of refined sugar to Frederick William III, of Prussia, in 1799. About ten years later, Napoleon used extraordinary efforts to foster the production of native-grown sugar, and grapes, plums, maize, sorghum, carrots, etc. were also experimented on. The results obtained did not excel those from beets, and the first French factory for making beet sugar was established at Lille, in 1810, by Crespel-Delisse. The sudden and great fall in the price of sugar, caused by the peace in 1815, crippled the native industry in Germany as well as in France, but a few of the manufacturers held on tenaciously, and the production of beet sugar, in France alone, rose through many vicissitudes from 1,000 tons in 1827 to 486,000 in 1887, and in still greater proportions in Germany.

5. The artificial conversion of starch into glucose was first accomplished by Kirchoff, of St. Petersburg, Russia, in 1702. Of late years, this industry has assumed important dimensions in Europe as well as in the United States.

As to the history of the other sugars, obtained from the maple, sorghum, and various palms, nothing definite is known. The preparation of sugar or sirup from green maize stalks is due to the ancient Mexicans, and has been carried on with varying success in this country and Southern Europe.

In the United States, up to 1889, the sugar industry was confined almost exclusively to the manufacture of sugar from sugar cane and refining of this and imported raw beet sugar. Through the efforts of Dr. William McMurtoie, Dr. H. W. Wiley, Chemist of the United States Department of Agriculture, E. H. Dyer, and others, the beet-sugar industry has steadily grown and bids fair to attain enormous proportions.

SUGAR CANE AND ITS COMPOSITION

6. The Plant.—The sugar cane, *saccharum officinalis*, is a gigantic grass; its stalk is round, knotted, and hollow, and the exterior is of a greenish-yellow or blue color, with sometimes violet streaks. It grows from 2.6 to 6.6 meters high and from 4 to 6 centimeters thick; its leaves are ribbed and vary in length from 1.6 to 2 meters. Most botanists are inclined to ascribe all varieties of sugar cane now grown to a single species, which, as has already been mentioned, is called *saccharum officinalis*. This theory supposes that all the forms which are at present met with are merely varieties induced by the effects of cultivation. The best authorities are not absolutely agreed on the subject, however; and as it is very questionable whether any of the canes now to be discovered growing in an apparently wild state in some of the oldest sugar-raising countries are truly wild, that is, have never been subjected to artificial influences, it is probably impossible to arrive at a reliable decision, especially since the original home, or homes, of the sugar cane remains unknown. This plant is grown from seeds and also cultivated from cuttings.

7. Components of the Cane Juice.—Under ordinary conditions, the nature of the ingredients composing cane juice is not liable to great variations, but the proportions of these ingredients fluctuate with the soil and climate, the age of the cane, the portion of the cane affording the juice, and other circumstances. Consequently, only an average composition of this juice can be given; it may be said, however, in general terms, that cane juice consists of about 81 per cent. of water, 18 per cent. of sugar, .6 per cent. of organic matter, and .4 per cent. of mineral matter; and further, that about .5 to .6 per cent. of the sugar in the juice of ripe canes, and still more in unripe canes, is uncrystallizable.

Although these constituents are very intimately combined in the juice, it is a well-known fact that the juice is not of a constant composition throughout the entire cane, and this fact is well recognized by planters, since they cut off and reject the tops of the canes before extracting the juice. Further it is to be noted that the juices contained in the soft central, or medullary, part of the cane are much more rich in sugar than those of the nodular portion. Conversely, it is found that the saline and organic matters are in increasing proportions in the harder parts of the cane. These are very important facts. The most saccharine (sugar-giving) juice is found in the softest parts of the cane, and is therefore most easily extracted; when an extra yield of juice is obtained by the exhaustion of the harder parts, the quantity is unfortunately at the expense of the quality.

8. Crystallizable and Uncrystallizable Sugar. — It has been stated that the sugar in the juice contains from .5 to .6 per cent. of uncrystallizable sugar. The relations between the two kinds of sugar is still an open question, and the conversion of uncrystallizable sugar into crystallizable sugar appears an impossibility; but crystallizable sugar is readily converted into uncrystallizable sugar. Bearing in mind that the crystallizable sugar is the one that is desired, such conditions that might help to invert the crystallizable sugar have to be avoided.

In the first place, the uncrystallizable sugar itself is a direct loss, that is to say, it has no value as sugar; and, in the second place, the existence of this uncrystallizable sugar in the sirup so affects the remainder as to greatly hinder, if it does not absolutely prevent, the recovery of an equal quantity of the still unaltered crystallizable sugar in a salable form. The reason for this lies in the fact that the liquid containing the altered sugar has a treacly consistency, and cannot be conveniently deprived of its water by evaporation to such a degree as will leave the unaltered sugar in a saturated solution capable of clean crystallization on cooling. Practically, therefore, in round numbers it may be said that every pound of sugar rendered uncrystallizable in a sirup entails an actual loss of 2 pounds of crystallizable sugar.

9. The chief alteration in sugar may be ascribed to the fermentation of the juice, or, perhaps more correctly, of certain parts of the juice, namely, organic matter other than the sugar. The conditions favoring this fermentation are free access of air to the juice and the prevalence of an elevated temperature. These are, however, natural conditions, and fermentation may already begin in the living cane, when injuries (such as the gnawing of rats, etc.) admit air into the plant cells. Artificial fermentation is set up in the juice the moment the latter is extracted from the cane, and is maintained by the heat necessary for carrying on the various processes of manufacture, increasing in proportion to the duration of the processes and to the degree of heat applied. Acids also favor fermentation; they are nearly always present in a free state in the juice, as is shown by the latter giving an acid reaction with litmus paper. Hence, the importance of rapid treatment at as low a temperature as possible, and the necessity of avoiding unnecessary exposure to air.

10. Mineral Matter in the Juice.—It is generally conceded that the mineral matter in the juice of canes best fitted for sugar manufacture amounts to about .429 per cent. of the liquid. But it appears that the nature of the soil in which the cane is grown influences the amount of mineral matter to a considerable extent; besides, this mineral matter is not evenly divided throughout the entire plant, as it is found in greater proportions in the head than in other parts of the cane.

The mineral matter contained in cane juice consists principally of potassium, sodium, calcium, and iron, these elements being present in the form of oxides, carbonates, chlorides, sulphates, etc.

11. Organic Matter in the Juice.—The organic matter in the juice, exclusive of the sugar, may be divided into three groups, namely, *granular matter*, *albuminous matter*, capable of coagulation by heat, and *nitrogenous matter*, capable of coagulation only by alcohol and metallic solutions. Their percentage proportion of the juice is, approximately,

Granular matter	.100%
Albuminous matter	.027%
Other vegetable matter	.223%

12. The **granular matter** is supposed to be found as corpuscles, or granules, suspended throughout the liquid, and consisting of globular, transparent bodies, whose outer skins contain a semi-fluid matter. This granular matter causes an appearance of milkiness in the liquid, and is only precipitated with great difficulties on standing, but may be readily, although not completely, separated by filtration. It appears that this granulated matter is bacteriologically connected with the fermentation of the juice, as a filtered juice may, under favorable conditions of temperature, be kept from 6 to 24 hours without fermentation taking place, although after this period, which varies with the temperature, the liquid becomes cloudy, corpuscles are developed, and fermentation sets in again; and at 25° C., well-formed bubbles of carbon dioxide appear in the liquid. On the other hand, when the juice has been simply cleared from the coarse fragments of vegetable matter, fermentation rapidly sets in after extraction from the cane and the liquid becomes viscid after a few hours.

At the boiling point, cane juice is freed from part of the albuminous substance that it contains, and this substance, coagulating under the influence of heat, seizes on the granular matter and draws it into flakes, which form on the surface of the liquid. This albuminous matter plays an important part in the fermentation of the juice. After being boiled, and thus becoming sterile, juice that has been completely freed from its albumen and its globules by means of filtration may be kept perfectly fresh for many hours at a temperature of 30° C.

From what has been said, it appears to be sufficient to raise the freshly extracted juice to its boiling point and to filter it immediately, in order to have a perfectly limpid liquid that can be kept for a considerable time without any alteration. Further, the globular and albuminous substances help to create acidity in the juice, and thus become responsible for the production of uncrystallizable, and the diminution of crystallizable, sugar. When these substances are eliminated, the production of acidity by the action of the heat is decreased, and always remains inferior to what it would have been in the contrary case.

13. The **albuminous matter** in the juice coagulates at about 80° C., and is precipitated by strong acids without being dissolved to any noticeable degree by an excess of the reagent.

After the albumen is partially separated by heat, there remains in the juice a complex organic matter that can be precipitated by either alcohol or neutral acetate of lead, and that is very soluble in acids as well as in alkalies. Separated and purified by repeated precipitation in alcohol, this substance, which is odorless, tasteless, and without influence on polarized light, when heated with calcium or potassium compounds, evolves ammonia. Mixed with either pure or sweetened water, it forms a disturbed and viscid solution, and it appears to be the real cause of that viscid consistence which cane juice and sirup assumes under fermentation. This substance, escaping the action of the agents used to defecate the juice, accumulates in considerable quantity in the sirups. It must therefore be considered as one of the chief causes that hinder the extraction of the sugar at the second boiling, as it is a powerful obstacle to the regular crystallization of this substance and excites rapid fermentation when sufficient water is present.

THE MANUFACTURE OF CANE SUGAR

14. Synopsis of Sugar Manufacture.—The various stages of the manufacture of sugar from sugar cane may be described as follows: (1) The extraction of the juice and its inherent constituents from the cane; (2) the separation from the juice of all the matters except sugar and water (known as *defecation* and *clarification*); (3) the removal of the water from the sugar (known as *concentration* and *granulation*); (4) the cleansing of the sugar crystals by washing and draining (known as *curing*).

EXTRACTION OF THE JUICE

15. The sugar-containing juice existing in the plant is enclosed in little cells, which are surrounded and protected by lignose or woody matter, the latter forming about $\frac{1}{10}$ of the total weight of the case. The liberation of the imprisoned juice may be affected either by (1) rupturing the cells so that the contents flow out, (2) combining a soaking in water with the rupturing process, or (3) utilizing the membranes of the cells themselves as a means of allowing the escape of the sugar and other salts in solution, by the process known as *diffusion*.

16. Crushing the Cane.—It may be truly said of cane mills that their name is legion, and a comprehensive account of all the forms introduced or proposed would fill a large volume. For all practical purposes, however, it will suffice to describe the typical arrangement adopted by nearly all engineering firms. It may be premised that no subject seems to be in a less satisfactory state, scarcely any two opinions coinciding as to what is the best form of mill.

The sugar cane is crushed in a press consisting of three hollow cast-iron rollers *a*, *b*, *c*, Fig. 1, placed horizontally in a cast-iron frame. By means of the screws *i*, *i*, the approximate distance of the rollers is adjusted. One roller is half as large again as the others, and is moved by three cog wheels, not shown in the figure, fitted on the axes of the rollers.

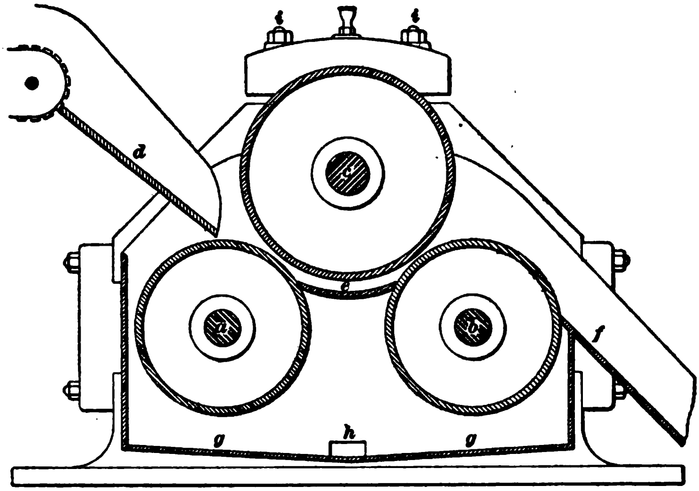

FIG. 1

The sugar cane is transferred from the slate gutter *d* to the rollers *a*, *c*, which press it a little, and from thence it is carried over the arched plate *e* to the rollers *b*, *c*. The pressed sugar cane falls over the gutter *f*, the expressed juice collecting in *g*, *g* and running off through *h*. The middle roller is termed the *king roller;* the side cylinders are individually the *side roller* and *macasse roller.*

Cane mills have been constructed with four and even nine rollers. In the four-roller mill, where two rollers are placed above and two below, the driving power required is naturally

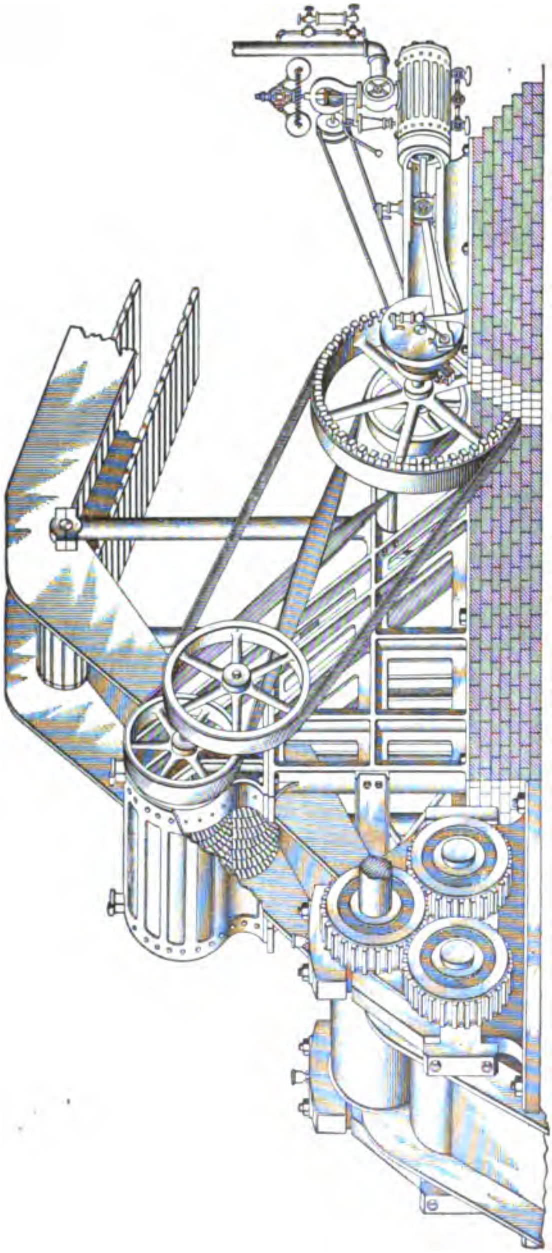

FIG. 2

somewhat greater than that required for an ordinary three-roller mill, but it is claimed that the yield of the juice is proportionally much larger. In the five-roller mill, three rollers are placed below and two above; 10 per cent. more juice is said to be extracted by this plant, but considerably more power is needed, and the bagasse (the extracted cane) is generally more broken up. In the ordinary three-roller mill, two pressures are supposed to be exerted in crushing, but in many cases the side roller is little more than a feeder for the canes, thus leaving the entire grinding to be virtually done by the macasse roller. A recent addition to cane mills is the application of hydraulic pressure to the rolls, so that the mill can adjust itself to the inequalities of feeding that appear to be unavoidable.

17. Shredding and Crushing the Cane.—The imperfect liberation of cane juice by crushing with the ordinary mill has led to experiments in other directions. A recent addition to the cane mill is a so-called *shredder*. In Fig. 2 is shown the *national shredder*, manufactured by the Newell Universal Mill Company, of New York.

The operation is readily understood and needs no further explanation. The advantages of such a combination of shredder and mill are many. The use of the shredder augments the quantity of cane the mill can grind, and it increases the extraction of juice and, consequently, the yield of sugar. It serves as a safeguard, making known at once the presence of any piece of iron, stakes from cars, or other substances that occur in the cane, which, by an attached automatic device, are allowed to pass through the shredder, thus giving ample time to remove them before any damage is done to the rollers. The shredder tears the cane into shreds of varying length, perfectly opening it, breaks the joints thoroughly, and forms a pulpy mass, which is the most advantageous condition to allow the mill to thoroughly press out all the juice without requiring the immense extra power necessary where the mill alone is grinding or crushing the cane. The shredder spreads the shredded cane uniformly and evenly the entire length of the mill rolls, and does away with the necessity of spreading the bagasse by hand between the mills, where regrinding is in use.

18. Fuel and Furnaces.—The use of coal and wood as fuel needs no remarks, depending simply on the cheapest supply. Whether it is more profitable to use the bagasse as either fuel or fertilizer is an open and much-discussed question, but as bagasse continues to be quite extensively used as fuel, it will be appropriate to give some figures concerning its evaporating power.

Assuming the cane mills to express 68 per cent. of juice, which, with modern milling machinery, is a modest estimate, 6,000 gallons of juice per hour would produce 30,325 pounds of wet bagasse. From experiments made on a large scale in Egypt, it appears that dry bagasse, fit for burning in furnaces ordinarily using it, weighs 53 per cent. of the wet; and 29,578 pounds of dry bagasse will evaporate as much as 16,000 pounds of ordinary coal, so that it requires 1.85 pounds of bagasse to do an equal amount of work as 1 pound of coal. Cane that yields 6,000 gallons of juice produces 16,072 pounds of dry bagasse, which quantity is consumed in the evaporation of 109.5 cubic feet of water, or, approximately, 20 pounds of bagasse to 1 cubic foot of water. As an approximation, 1 pound of coal is equal to about 2 pounds of dry bagasse, so there seems to be margin enough to warrant the statement that the refuse of the canes should give sufficient fuel to make the sugar, especially where the climate is favorable to drying the bagasse.

Bagasse, however, whether wet or dry, cannot be burned in an ordinary furnace, owing to the fact that from 26 to 54 per cent. of the ash is silica, which quickly forms a deposit upon the bars of an ordinary furnace. A number of suitable furnaces have been constructed for this purpose. The drying of bagasse requires a certain amount of labor and involves expense. About 10 years ago, J. L. Marie, of St. Pierre, Martinique, invented a furnace in which bagasse can be burned in the condition that it leaves the mill.

Figs. 3 and 4 represent a longitudinal vertical section and horizontal section, respectively, of a wet-bagasse burner. The furnace chamber *a* is constructed preferably of cast-iron plates and is stiffened by iron ribs *b*, which are bolted together by flanges *c* and encased in brickwork. The pyramidal crown *d* of the furnace chamber is also constructed of cast-iron plates, bolted upon chamber *a* and surmounted by the hopper *e*, in which the bagasse is dried and through which it is fed to the furnace. A self-acting balance door *f* is placed within the hopper in the inclined position shown; it works on pivots at *g*, supported in the sides of the hopper, upon which is placed a counterweight *h*, adjustable along the arm *i*, to regulate the quantity of bagasse admitted each time the door opens. The fire-bars *j* at the lower part of the furnace are inclined as shown, and their lower ends extend through an opening *k* and are supported by an inclined bridge *l* bolted to extensions *m* of the side walls of the furnace. The upper part of the opening is surmounted by a flange *n*, which may fit in the doorway or beneath the fire-box of the boiler, the form and dimension of this flanged opening being varied as circumstances may require. The door *o* just above the fire-bars gives access to the furnace.

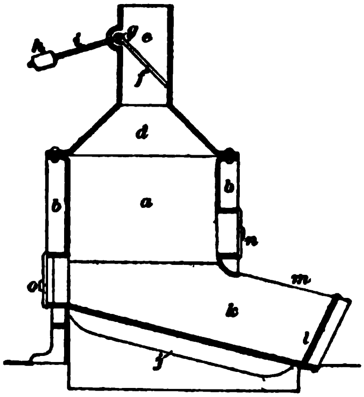

FIG. 3

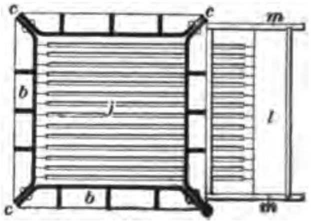

FIG. 4

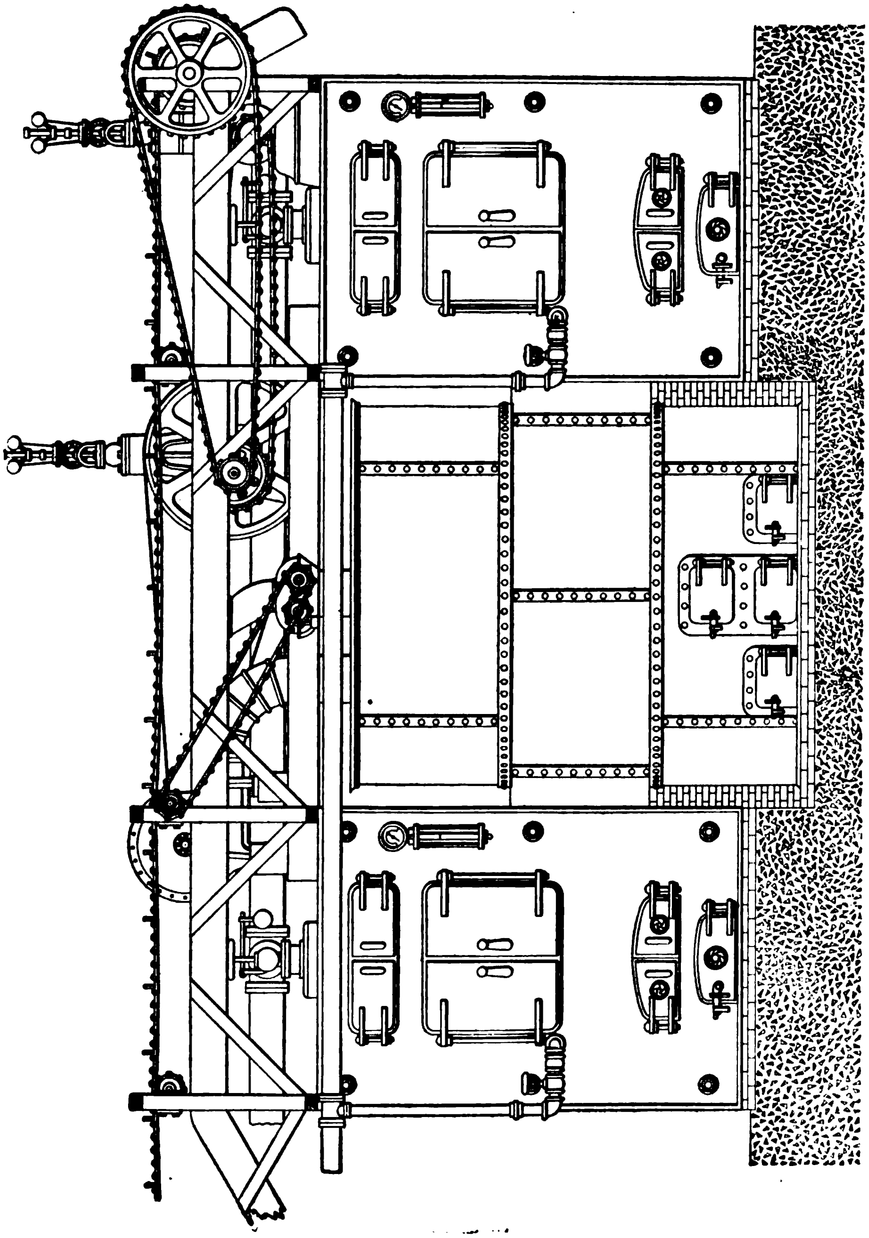

Fig. 5

19. A similar burner connected with multitubular boiler is shown in Fig. 5. This burner, which is manufactured by the Newell Universal Mill Company, of New York, is circular, and the bagasse as it is fed into the top of the burner falls upon the grate in a round conical pile, covering the entire grate. Thus the air has to pass through the pile of burning bagasse, creating thorough combustion of the gases in the chamber, thereby enabling the boilers to utilize the heat with a minimum of escape at the chimney. The bagasse is conveyed from the mill by a drag carrier to the tops of the burners and fed through adjustable discharge gates into hoppers, which automatically deliver to each burner just the amount that is needed. The material in excess of that required for the first burner passes on to the next, and so on in the same manner. Any surplus bagasse is carted away and stored for future use.

The advantages of a wet-bagasse burner are not merely confined to the saving of labor and time, but extend to the equally important gain represented by the avoidance of that risk of fire which is so constantly to be feared when large quantities of dried bagasse are stored away, and by the utilization of the combustible quantities of the saccharine matter left in the canes, before it is destroyed by fermentation.

20. Maceration.—It has been sought to facilitate the extraction of the juice from the cane by submitting the cane to the action of water or steam, either before the crushing operation in the roller mill or at an intermediate stage between two such crushings. Several methods have been devised for carrying out the saturating process on a practical scale, among the most important being Duchassaing's *maceration*, or *imbibition*, *method.*

The apparatus employed in this method is shown in Fig. 6. The working of the process will be readily understood from the figure and the following description: The mill *a* receives the cane and crushes it in the regular way, giving approximately 68 per cent. of juice. The bagasse

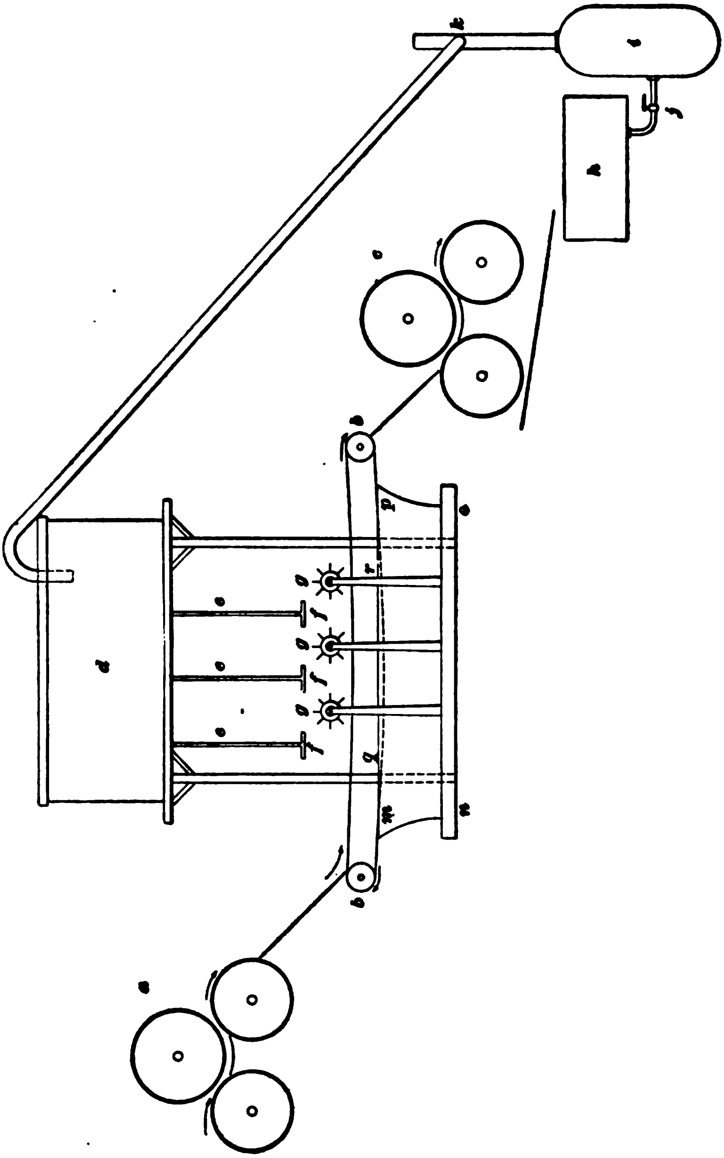
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FIG. 6

falls upon an endless cloth *b*, which conducts it to a second mill *c*; *d* is a tank containing boiling water; *e* are tubes terminating in pipes *f* parallel to the endless cloth, which sprinkle water from tank *d* upon the bagasse passing from the first to the second mill; *g* are beaters that turn the bagasse, and thus equalize the imbibition; *h* is another tank, which receives the juice from the mill *c*; *i* is a monte-jus, which sends the juice, if its density is not sufficiently great, into the tank *d*, to serve for a second maceration of new bagasse; or, if it is dense enough, by the joint *k* to the defecation department. The endless cloth *b* dips so that the portions between *q* and *r* immerse the bagasse in boiling water contained in the vessel *m n o p*, thus increasing maceration. Since the apparatus has come into extensive use, it has been simplified by dispensing with the beaters *g* and the vessel *m n o p*.

21. Maceration by Russell and Risien's Process.—In this process, which is similar to Duchassaing's process, two cane mills are employed. They are connected by an intermediate chamber, in which runs an endless band, or chain, which serves as carrier of the partially exhausted bagasse from one mill to the other.

The mills are placed at a convenient distance from each other, and the chamber connecting them is in the form of a chute, or trough. Inside the chamber, near its top, is placed a system of perforated pipes, by which either hot water, cold water, or cane juice may be sprayed upon the partially exhausted bagasse. Between the carrier is placed a second series of perforated pipes, which are used to drive steam through the carrier, and mixing with the water or cane juice, saturate the partially exhausted bagasse. The juice from the first mill, after passing through a sulphurous-gas churn, is forced through a set of combined juice heaters and water traps, and the condensed water and steam from the water traps is conducted to the piping described at the top of the chambers for saturating purposes. The bagasse, being conducted from the first mill to the second by the endless band, is thoroughly saturated by this condensed water, and is further treated by steam from the lower pipes passing up through the band, which steam also serves to cleanse the band. When the bagasse reaches the second mill, the water that has been absorbed by saturation is expressed from it. This water carries with it the soluble matter contained in the canes. The juice obtained by this second treatment is carried through a separate set of juice heaters, whereafter it may be mixed with the juice from the first mill, or it may be conducted into the piping in the chamber and used for saturating the bagasse as it circulates, thus absorbing a larger proportion of saccharine matter; it is eventually conducted, when of sufficient strength, to augment the supply from the first mill.

22. Diffusion.—During the past few years, the process of diffusion has been employed in some of the cane-sugar works, especially those located in this country, but as this process originated in the beet-sugar industry, and is exclusively used as the means of extracting the juice from beets, it will not be treated here, and the student is referred to the section on the manufacture of beet sugar for information.

DEFECATION AND CLARIFICATION

23. After the juice has been extracted in one way or the other, it becomes necessary to remove everything that contaminates the juice, as it is evident that in order to obtain sugar that is as pure as possible and readily crystallizes, nothing but the sugar held in aqueous solution is desirable. The amount of impurities varies very much according to the method of extraction used, the juice obtained by diffusion being comparatively the purest.

24. Preliminary Straining.—The juice obtained by milling, or maceration, always contains a considerable amount of impurities, which are most easily eliminated by passing the juice through one or, preferably, a series of strainers. One of the most efficient contrivances for straining the juices is an endless wire-web strainer. The straining web is of fine-wire gauze and revolves on three horizontal rollers, two of which are on the same level as the upper angles of a V, the third being at the lower angle and immersed in a vessel with water. A scraper is set closely to the web, just after it has turned the roller on its descent, which, in connection with the water through which the web passes, keeps it clean. The strained juice is received in a tray placed immediately under the horizontal part of the strainer, and passes from thence to the clarifier.

The chief agencies hitherto introduced to effect the cleansing of the juice are heat, chemical action, and sometimes filtration. Their application will now be considered.

25. Heat.—As has been previously stated, heat alone exercises considerable beneficial effect in checking acidity, that is to say, scalding the juice prevents acetous fermentation setting in, probably by destroying the particular fungous germs that are the cause of that fermentation, also, by evaporation, a portion of acids holding the albuminous matters in solution are driven off, whereby the albumen is coagulated and rendered insoluble. But steam is rendered most valuable as an aid to the action of chemical preparations upon the juice, increasing the energy of the reactions set up, and thus greatly reducing the duration of the operation.

26. Defecation by Means of Chemical Reagents. The word *defecation* implies the removal of the fecular matter, or, practically, the breaking up of the albuminous compounds. As we have already seen, this is effected to a certain extent by heat, which evaporates a part of the acids holding the albuminous matters in solution and whereby the albumen is coagulated and rendered insoluble. The result thus obtained, however, is much more completely accomplished by the simultaneous application of a strong alkaline earth, such as lime, which combines with the liberated acids. It must be remembered, however, that any excess of lime beyond that which is required to neutralize these acids will redissolve the coagulated albumen and keep it in the state of solution until this excess of lime is again neutralized by the addition of an acid. The operation is obviously a delicate one, demanding constant supervision.

27. The first point to be ascertained is the exact amount of lime required to neutralize a given quantity of the juice. For this purpose, a definite amount is measured out, heated to boiling, and titrated hot with a standard calcium solution, using either litmus paper as an outside indicator or adding either a little extract of logwood or a solution of methyl orange.

When the point of neutralization has been reached, the beaker containing the neutralized sugar solution is removed from the flame and its contents allowed to settle for a minute. If a coagulum consisting of rather large flakes is seen floating about in the transparent but somewhat colored liquid, which separate and settle readily to the bottom of the beaker, the point of proper clarification has been reached—that is to say, in such a case, the point of neutralization and clarification coincide. If, however, on the other hand, the flakes are small and do not readily separate from the juice, and the liquid is not perfectly transparent, it is not in a condition to filter readily, and on boiling would throw up a scum, and thus lead to a loss of juice.

The beaker is again placed over the lamp, its contents is brought to boiling, and more of the standard solution is added, in small portions at a time, until the indications of proper clarification, as above described, are obtained. The strength of the calcium solution is unimportant as long as its strength is positively known.

28. Defecation and Clarification by Lime. — The juice being tested as to its density by means of a hydrometer, and its acidity by means of titration with a calcium solution, and the milk of lime of proper strength being prepared, the twin process of defecation and clarification may commence. There are several ways of carrying it into operation. About the most simple, and one that is practiced quite frequently, is known as *cracking*. It necessitates the use of two or more clarifiers, and is conducted as follows:

The strained juice is admitted into the clarifier until a sufficient amount has accumulated to prevent any injury by heat. Fire is then placed under the clarifier (or steam is admitted into the jacket or coil), and by the time it is full of liquor the temperature will have risen considerably, probably to about 54° C. (130° F.). The lime is then added, thoroughly incorporated, and the heating is continued. A thick greenish-yellow scum soon appears on the surface and rapidly increases in thickness, changing color at the same time from exposure to the air; as the temperature approaches the boiling point and is heated up to about 79° to 82° C. (174° to 180° F.), numerous minute air bubbles are seen to rise and form a frothy layer under the thick scum. By and by these air bubbles force their way, at a few points, through the dark, dirty-looking scum, which soon cracks in several places, and the white frothy bubbles appear in the cracks. When this point has been attained, the heat is quickly withdrawn and the contents of the clarifier are allowed to rest for 15 to 30 minutes or more. Ebullition is carefully avoided, because it would break up the floating scum and diffuse it through the mass of the liquor.

The time allowed for settling depends on a variety of circumstances—the nature of the juice, the proper apportioning of the lime, and the time that can be allowed consistent with getting through a good day's work. After settling, a layer of coagulum is still found at the top, and another layer at the bottom, while the great body of the liquor is tolerably bright and transparent, although, perhaps, more or less colored and with a quantity of minute flakes floating thickly in it. If it is hazy from minute, generally diffused, solid particles, the operation is incomplete, and either the heat has not been sufficient to clarify or the lime has not been used in adequate quantity. After standing for a length of time, depending on the original conditions of the juice, the clear liquor is run off into an evaporating apparatus; the scum and sediment, with the considerable quantity of juice that invariably accompanies them, are frequently carried to the skimming cistern, to be used in setting up liquor for the manufacture of rum.

When the clarifier has either a coil of steam pipe or a steam jacket, it is much more manageable, and in this case it is generally so arranged that little loss of time occurs, for as soon as there is enough liquor in the clarifier to render it safe, the steam is turned on in such measure as to attain the desired temperature by the time the vessel is full of liquor. Fire clarifiers are generally discharged by a stop-cock near the bottom until the liquor begins to run muddy. Steam clarifiers are discharged by a valve in the bottom in connection with a tube that rises 4 to 6 inches above the bottom, so as to avoid disturbing the sediment as much as possible.

29. This method is open to many grave objections, the principal of which are the following: (1) That clarification is very rarely obtainable below the boiling point of the juice, the consequence of which is that the juice wants brilliancy and transparency, and has minute, light, floating particles which render the process of filtration not only extremely slow, but also unsatisfactory. (2) During the concentration, this finely divided floating matter is thrown up as scum, causing an unjustifiable waste of juice in the skimmings.

To overcome these drawbacks, Doctor Shier introduced the following modifications: The strained juice is admitted into a clarifier and boiled briskly for 5 minutes; the scum that rises is constantly beaten down by a wooden or wicker plunger. While thus boiling, the proper quantity of lime is added, this lime being mixed with a proportion of clay batter, gypsum, or whiting batter; the boiling is continued for a few minutes, with constant stirring and beating down the scum. Neutralization being effected, the whole contents of the clarifier are rapidly withdrawn into a subsider and left there until all the coagulated flocculent matter has subsided to the bottom of the vessel. The clear juice is drawn off without disturbing the sediment and passed through a filter into a cistern. Here any excess of lime that may have been used is corrected by the very careful addition of dilute sulphuric acid, the quantity of acid necessary being previously ascertained by the titration of a sample of the juice. Although it is most advantageous to have the juice exactly neutral, the safest way is to cease adding sulphuric acid when the alkaline reaction becomes extremely feeble, and thus avoid a possible excess of acid. If the lime is left in considerable excess, the sugar will be dark colored, and if there is an excess of acid, the grain will be fine and soft, and part of the sugar will be inverted to the uncrystallizable condition.

The reason for adding clay or other heavy matter to the lime is to produce a mechanical precipitation of the impurities, which may be filtered off, instead of having to deal with a scum that needs skimming, and causes a considerable loss of good juice.

30. The clay batter is best prepared by using any stiff adhesive clay containing little sand and coming from such a depth as to be free from roots and as much organic matter as possible. This clay should be well dried in the sun, crushed to powder, and squeezed through a wire-gauze sieve of 10 to 14 meshes to the inch. Clean water is put into any appropriate vessel, and the sifted clay is poured into it gradually, mixing it well until the whole is of the consistency of cream or batter. From 4 to 8 gallons of this batter, mixed with the ascertained quantity of cream of lime, should be sufficient to clarify 500 gallons of cane juice. When either gypsum or whiting is used in place of the clay, it must be in very fine powder.

31. Howard Process.—This process is strongly recommended. It may be described as follows: The juice is strained on its way to the clarifier and is then gently heated; for each 100 gallons of juice, 2 ounces of finely sifted quicklime is made into a cream with water and added to the clarifier; the whole is well stirred and the temperature is allowed to rise to 82° C. (180° F.), until a thick crust forms on the surface and shows a disposition to crack. As a rule, this will occupy from 15 to 20 minutes after the addition of the lime; if it is very slow in forming, the heat may be very slowly raised to 93° C. (200° F.), but *not* beyond. When the crust has formed and shows signs of cracking, the fire is stopped and the liquor is allowed to rest for 10 minutes, when, after expiration of this settling time, it is drawn off through a fine strainer into a second vessel, known as the *precipitator*.

Here the heating is again started until the liquor has reached as high a temperature as can be obtained without producing actual boiling. In the meantime, the rising scum is constantly skimmed off as long as it appears. After a short period of skimming, the liquor may then be boiled, continuing the skimming for 10 or 15 minutes. At the expiration of this time the skimmer is laid aside and *Howard's finings* are added. These finings are well stirred in, and the boiling is prolonged for another 2 or 3 minutes, when the whole is thoroughly agitated and quickly run off into a fining cistern, or subsiding tank, and allowed to rest for 2 to 6 hours before passing through charcoal filters into the evaporators.

Howard's finings are prepared in the following manner: Well-burned lime is slaked with boiling water, so as to form a "cream"; to this is added an equal bulk of water, and the mixture is boiled until the lime assumes the appearance of fine curd; the extraneous matter is then washed away, and the lime and liquor are run through a fine sieve. The next part of the process is to dissolve in 6 gallons of water about 2½ pounds of alum for every 100 gallons of cane liquor that is to be refined, adding to such solution about 3 ounces of whiting (purified chalk) for each 3½ pounds of alum, the mixture being stirred until effervescence ceases. It is then allowed to subside, and the solution (containing mostly potassium sulphate, which is very injurious to sugar) is drawn from the precipitated matters, which are alumina and calcium sulphate. After this the precipitate is put with the prepared lime curds and shaken up with the water they contain, the whole being agitated during the effusion. The curds are to be in such proportion that paper stained with tumeric shall barely change its color by immersion in the mixture, and shall recover its former yellowness when dry.

The finings being thus carefully prepared are allowed to settle to the bottom of the vessels in which they are contained; and after draining off the supernatant liquor are placed upon blankets, supported in the manner of a filter, and the moisture is drained off until the mass begins to contract and cracks on its surface; the finings are then fit for the clarification of the liquor. Addition of cane liquor to them is made in such proportion as will bring it to a creamy state, and then the whole is mixed equally into the cane liquor to be fined. The clarified cane liquor is allowed to remain for several hours before the bright liquor is drawn off from the finings. The object is to procure aluminum sulphate free from potash and ammonia. The alumina greatly assists the purifying action of the lime.

32. Calcium Bisulphite.—The bleaching and cleansing action of sulphurous acid led to experiments on its applicability to the defecation of cane juice, and the first form in which it was employed was as a compound with lime, known as calcium bisulphite. One of the most successful methods of using it has been adopted by Doctor Shier, in British Guiana, and is as follows: About 1 per cent. or even less of solution of calcium bisulphite is added to the juice as soon as possible after it is extracted, or even while it is being extracted. Heat is then applied, and after the juice has been boiled and stirred for a few minutes, a mixture of cream of lime and clay batter is added. The exact quantity of cream of lime is ascertained by a preliminary test, as described previously, sufficient only being used to produce actual neutrality. After boiling for 5 to 10 minutes, and beating down the scum, the contents of the clarifier are run into a subsiding vessel and thence filtered out for concentration. The subsidence is not efficient without the addition of clay batter or some similar matter that acts purely mechanically, but the sirup thus obtained has a very fine color, and gives a superior-looking muscovado sugar. One of the chief objections to its use is the high price of the calcium bisulphite.

33. Defecation by Sulphurous Acid and Lime.—The process of introducing lime and sulphurous acid into the juice separately has found wide use in this country, the West Indies, Egypt, and other places. There are two different methods of carrying this out—namely, by first passing the sulphurous acid into the juice and then adding lime, and secondly, by first adding the lime and then introducing the sulphurous acid. The effect of both methods is probably identical, but the first method is more generally adopted and will be selected for description.

34. As fast as the raw-juice tank is filled, its contents is raised into the so-called clarifiers, steam at a pressure of about 60 pounds per square inch being turned on as soon as the copper bottom of the clarifier is covered by the juice. When the juice begins to boil, it is stirred with a copper pipe, through the lower perforated end of which sulphurous-acid gas is injected and allowed to dissolve in the juice until the color of the latter becomes considerably lighter and a decided separation of the flocculent matter is seen to take place. The proper quantity of sulphurous acid to be added varies not only with the state of the cane, but also with the conditions of weather, etc., and can only be determined by practice, but it may be stated that a clarifier of 450 gallons capacity approximately requires the combustion of from .1 to .5 pound of sulphur.

The sulphurous acid is forced into the juice by means of a pump driven by a small engine, the speed of which can be regulated according to the quantity of gas required. The sulphurous-acid gas is generated by the combustion of crude sulphur in an oven, the air necessary for the combustion being sucked through the pump. As the combustion depends on the supply of air and the latter on the speed of the pump, the whole apparatus is actually self-adjusting.

As soon as the juice is sufficiently gassed, milk of lime mixed with china clay is added, until the juice is neutralized, from ½ to 3 gallons per clarifier of a capacity of 450 gallons of juice being usually required. The liquid is then let out by cocks in the bottom of the clarifiers into subsiders, where it is allowed to rest until the impurities have settled down, when it is decanted, by means of sliding overflows, into the clarified-juice tank. After the juice is properly clarified, it is perfectly clear and has a light-yellow color. The process of clarifying and subsiding takes from 1 to 1½ hours.

35. The lime used is the ordinary product of native limestone; it is mixed in two circular tanks fitted with agitators. The contents of one of these tanks is allowed to subside, so as to yield clear lime water, which is used in washing down the cane mills, juice gutters, and pipes. In the other tank, the milk of lime, which should have a density of about 9° Baumé, is prepared by mixing 9½ parts of cold water with 1 part of lime, to which an equal weight of china clay is added. The function of the latter is to assist mechanically in carrying down the impurities of the juice.

The scum that collects in the bottom of the subsider is removed by means of valves and runs down gutters to either of two tanks, from which it is filled into bags and placed in filter presses, described in the manufacture of beet sugar, whereby the juice is removed from the scum. The solid scum forms about 5 per cent. of the raw juice. The clear, pressed-out juice is pumped at once into the clarified-juice tank and the solid refuse is usually discarded. The subsiders are washed down by means of a hose at the end of each operation, the foul water being run off through wash-out valves and pipes.

36. Clarifiers having a capacity of 450 gallons of cold juice are about 2 feet deep and have a diameter of 6.5 feet.

They consist of copper pans 1½ feet deep, bolted into cast-iron steam jackets, and are surmounted by galvanized-iron cylinders, 1½ feet deep, in which skimming overflows, 2 feet wide, are formed. The heating surface of each is 52.58 square feet. Steam at 60 pounds pressure is admitted by 2½-inch valves, and the condensed steam is taken off by self-acting traps, one to each clarifier. The juice is let out by 4-inch cocks, worked by levers placed beyond the hand rail over the subsiders; and ½-inch petcocks, to ascertain the state of the steam jacket and let out any air, complete the equipment.

37. Steam is turned on as soon as the copper bottoms are covered with juice, and the latter, usually having a temperature of about 22° C. when pumped, begins to boil in about 20 minutes, and is kept boiling for about 5 minutes. A small portion of the impurities floats on the surface and is skimmed off at the lips provided for this purpose, whence the skimmings flow by suitable shoots to the tanks that receive the rest of the scum.

The subsiders correspond in number to the clarifiers. They are plain cast-iron tanks, 6 feet square and 2½ feet deep, with outside flanges and angles rounded to a 4-inch radius, for facility in cleaning. Each tank is provided with an overflow, actuated by screw and hand wheel, for the purpose of decanting the clear liquid, which is discharged into an iron tank running across the mill under all the subsiders; while for the discharge of the scum and for the subsequent washing, two plugs are provided, one of which is connected with the scum gutter and the other with the waste pipe. It requires about ½ hour for the juice to subside.

38. The Generation of Sulphurous Acid.—The sulphurous-acid gas is produced by the combustion of sulphur in a cast-iron D-shaped muffle, provided with a grate below, and the air supply is regulated by a sliding door at an end of the muffle. A 3-inch cast-iron cooling pipe, provided with numerous doors for cleaning and removing any sulphur that might have distilled over, conducts the generated gas to two double-acting pumps that are driven by an independent engine so that their speed can be readily regulated. From the pumps, the gas is led into a receiver, and from there into a 3-inch distributing pipe that runs full length under the clarifier stage, a 1-inch copper branch rising between each pair of clarifiers and terminating in a cock and rubber hose fitted with a copper stirring pipe, the extreme end of which is finely perforated, to allow a uniform distribution of the gas through the body of the juice in the clarifier.

39. The specific action of sulphurous acid, whether introduced, as has been here explained, in the gaseous form or liberated from a sulphur compound, is (1) to prevent fermentation of the juice, (2) to decolorize the juice, and (3) to cause a coagulation of those albuminous matters that are not affected by heat alone. They may be considered the advantages of this process. Its disadvantages are that the sulphurous acid is quite freely absorbed by the juice and that, even with the greatest care, an excess is readily applied, besides every precaution has to be exercised to prevent the oxidation of the sulphurous acid into sulphuric acid, the latter seriously interfering with the crystallization properties of the juice. It is therefore customary to add an excess of lime in order to insure the formation of calcium sulphate, should such an oxidation occur. By the combined use of lime and sulphurous acid, it has become possible to produce an almost white sugar.

CONCENTRATION AND GRANULATION

40. After the cane juice has been reduced to the condition of a solution of sugar in water, by the aid of one or the other of the processes previously described, the next step in the manufacture of sugar is to deprive this solution of that excess of water which would prevent the crystallization of sugar; or, in other words, the solution has to be concentrated to such a degree as to make crystallization possible.

Although the primary object of this process is to induce crystallization by disposing of the superfluous water, the student should bear in mind that it must also be indirectly a process of chemical and mechanical purification. By the act of crystallization, the particles of the substance to be crystallized come together to form a definite solid; they leave in solution those bodies that are present in too small proportion to admit of their crystallizing out as well as those that are incapable by themselves of crystallization. When freed from their mother liquor, the crystals must then be considerably purer than the solution from which they originated.

41. Referring to what has been stated previously, we know that a large number of chemical compounds crystallize; that is, they possess the property to assume a solid, definite shape out of a saturated or supersaturated solution on cooling. This property, as we have previously seen, is based on the power of water to hold compounds in solution in a degree varying with its temperature; this power, as a rule, increases with a rise in the temperature of the water and decreases as the same falls. If, for instance, a definite amount of water is made to dissolve as much sugar as it is capable of holding in solution at a given temperature, and this saturated solution is then allowed to cool slowly, the decreasing solvent power of the water forces the sugar to separate from the water in crystalline form. These crystals, which we know are composed of sugar and so-called water of constitution, are nevertheless considered as pure sugar, because the water is an essential necessity in the formation of those crystals and is chemically combined with the sugar, and consequently cannot be driven off without decomposing the sugar. The size of the crystals thus formed depends on the prevalent conditions, that is, the duration of the operation, the amount of the water originally present, and the agitation or quiescence of the liquor.

42. In reducing the sugar solution to a state of saturation, it is important to bear in mind the changes to which cane sugar is liable under the influence of heat. It first begins to melt. If the heating is then continued slowly and regularly, the sugar parts with successive molecules of water, becoming converted into a number of crystallizable, non-saccharine compounds, until, on further heating, it is converted into a dark-brown mass, known as *caramel.* As the operation of the solution proceeds, the mass thickens and the difficulty to impart equal heat to all parts of the mass increases with the consequent danger of overheating those parts next to the source of heat and converting the same into caramel. Another change that is constantly proceeding in the heated liquor is the inversion of crystallizable sugar into uncrystallizable sugar, the formation of acids and salts, this conversion being favored by the exposure of the hot mass to the air. The consequence of this inversion is *molasses*, which may generally be regarded as an artificial product of sugar, composed of uncrystallizable sugar, colored by caramel. The market price of molasses being much lower than that of sugar, it is evident that the prevention of this formation must be one of the chief aims of the sugar maker.

43. Open Pans Heated by Fire.—The earliest and crudest form of evaporating pans, but one that is still in operation, especially on colonial sugar estates abroad, is the "copper wall," or "battery of open pans," known as *teaches*, *taches*, etc. This is shown in Fig. 7.

The first two pans, *a* and *b*, of the series, are the clarifiers from which the juice flows into the taches *c*, *d*, *e*, *f*, which are simply copper vessels set in masonry on a descending plane; thus, as the juice becomes concentrated, each lower pan is filled with liquor from the one immediately above it until the liquor in the last pan *f*, known as the *striking pan*, is of such a density as to permit granulation, when the thick, crystallizable mass is ladled into shallow wooden vessels and removed to be cured. Originally, the liquor was ladled by hand through the entire series, but an improvement has been introduced, which consists of a dipper fitting into the striking tache *f*, and having at the bottom a large valve that opens upwards and works by a lever. The dipper is attached to a crane, the swing of which commands the striking tache and the gutter leading to the coolers. This arrangement, imperfect as it is, nevertheless economizes time. The furnace *g* for heating the series is set under the striking tache *f*, from which the heat passes by the flues *h* to the chimney *i*.

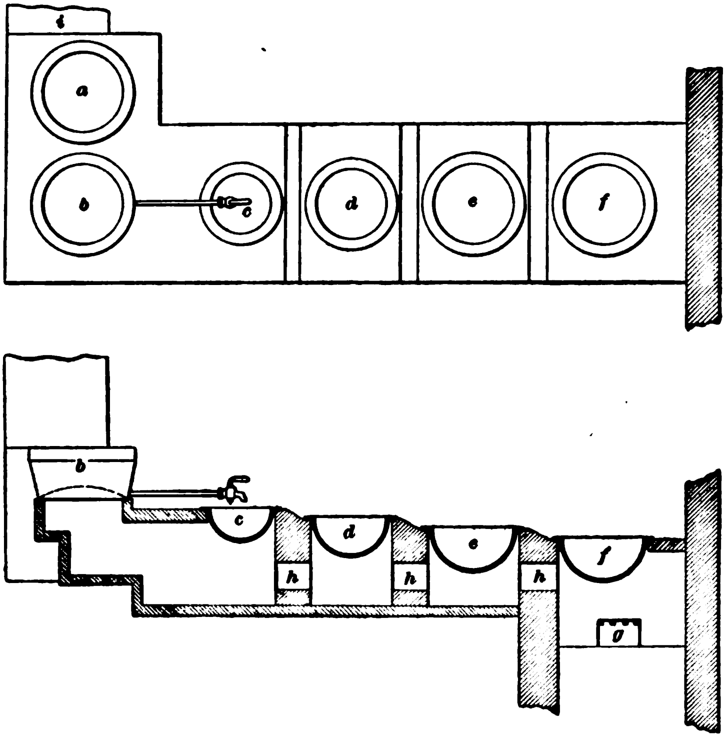

FIG. 7

Where such a battery is still in use, and this is mostly in the English colonies and other tropical countries, the chief difficulty encountered is the determination of the exact moment when the boiling of the liquor, technically known as *sling*, in the striking tache must cease, or in other words, the exact moment when to remove the sling to the coolers, or, as the sugar man calls it, "when to make the skip." Great skill and experience are required to decide the correct boiling time suitable for each kind of juice, as the main point is to bring the sling into such a condition that as great a mass of sugar as possible will crystallize out after it cools. If the sling is taken out of the tache too soon, there will be seen in it, after cooling, only a number of large irregular crystals, and a considerable quantity of sugar will be left in the molasses. On the other hand, if the sling has been allowed to boil too long, a sticky mixture of very small sugar crystals and sirup will result, from which the molasses cannot be drained off at all, or only with great difficulty, and from which it will be impossible to obtain clean, dry, and hard sugar crystals. An experienced operator, however, readily knows, from the way the solution is boiling, the approach of the right striking point, but if a test is wanted, a reliable and simple one is as follows:

Pour a spoonful of the boiling sling in a beaker containing clear water; if, after a minute's boiling, the sling can be formed into a ball that does not stick to the fingers, and that slightly flattens itself on the bottom of the glass by the mere weight of its descent on being dropped into it, the correct period for striking has arrived.

44. The only advantage of such a battery is the cheapness of its construction, while its disadvantages, on the other hand, are many. The process requires considerable fuel, and the finishing of the product requires a long time and depends too much on a skilled manipulator. Besides, the liquor in the pan is continually exposed to the action of the air while boiling, as well as in the process of ladling, which inverts too much of the sugar and produces an unreasonable amount of molasses.

45. Vacuum Pan.—The difficulty of boiling dense liquids is too well known to require more than passing notice. The cause of this difficulty is the lessened ability of the vaporized water to overcome the pressure of the atmosphere, which normally amounts to about 15 pounds per square inch; and it is readily understood that by relieving the liquid of this pressure, the boiling point is lowered and the water more easily evaporated off, thus reducing the amount of the fuel required and at the same time lessening the danger of producing caramel and molasses instead of sugar. To apply these principles to the concentration of sugar sirups, various forms of vacuum pans have been introduced, in all of which the boiling proceeds *in vacuo.*

Only the simplest form of vacuum pan will be described here, a description of the more complicated combination of three or four pans being given in the manufacture of beet sugar, the principles of which are the same.

46. Description of Single Vacuum Pan.—A vacuum pan consists of either a cast-iron or a copper pan, with dome cover and conical bottom, having a sharp fall towards the outlet. The heating surface consists of five seamless copper coils with brass inlet and outlet pipes, which are stayed to the sides and top of the pan with brass clips and rods. Each coil is supplied with steam from the boiler by either of two valves, one for low-pressure steam and the other for high-pressure steam. On the top of the pan is fitted a capital, with a manhole and cover, and a large vapor pipe, which leads to a save-all, where any watery particles carried over by the vapor are intercepted, and may be returned or withdrawn as desired. From the save-all, the vapor passes downwards into a condenser of extra-large capacity, supported from a staging, and fitted with a large injection pipe, which can be easily removed or examined by opening a manhole cover provided for that purpose. The condenser is connected by means of a conical outlet pipe to the pumping engine. The pan is fitted with two charging cocks for different qualities of sirup, proof stick, test cock, large oblong sight glasses with wash cups, steaming cock for cleaning the pan, barometer, thermometer, and air cock, and is lagged with wood, felt, and brass bands. At the bottom of the pan, a large outlet sluice valve is provided, especially arranged to prevent the *masse cuite* (condensed liquid) from adhering to the working surface. This valve is worked by a hand wheel and screw from the under side of the staging. The whole is supported on an iron stage, iron staircase, and strong cast-iron columns.

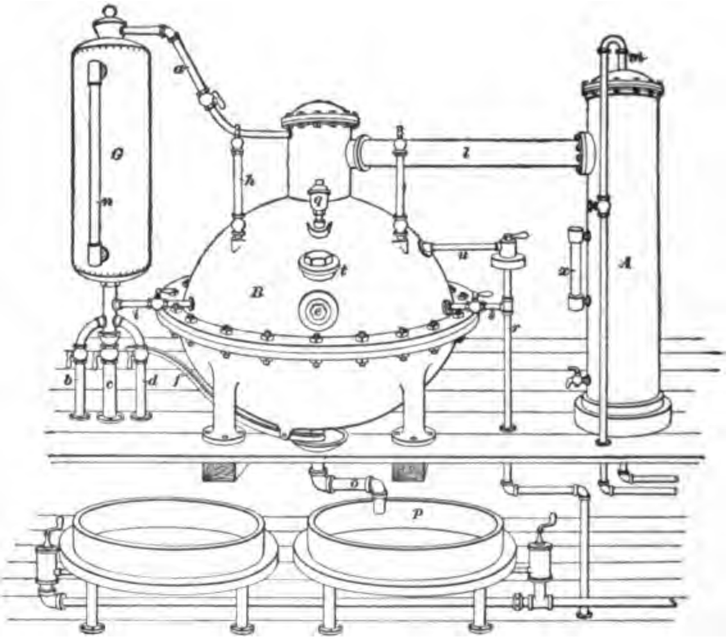

FIG. 8

A steam receiver is provided, to collect the exhaust steam from the vacuum pump and other engines in the factory for the supply of coils, and it is furnished with an escape valve, pressure gauge, and steam trap. The condensation water is withdrawn by means of a separate steam trap from each pan coil. The vacuum pumping engine is of unusually large capacity, to insure the maintenance of a very high vacuum and consequent efficiency of the pan. This vacuum pumping engine is especially designed to avoid several of the disadvantages inherent in most of these engines; the heavy flywheel is entirely carried between the bearings and the pump ram, and the steam piston is directly connected without the intervention of link work. The whole pump occupies very little of the usually valuable floor space, and all parts are easily accessible for examination and repairs. The pump is of the floating-arm-displacement type, with suction and delivery valves of very large area, and is so arranged that all the valves, grids, and guards can be withdrawn and replaced in short order.

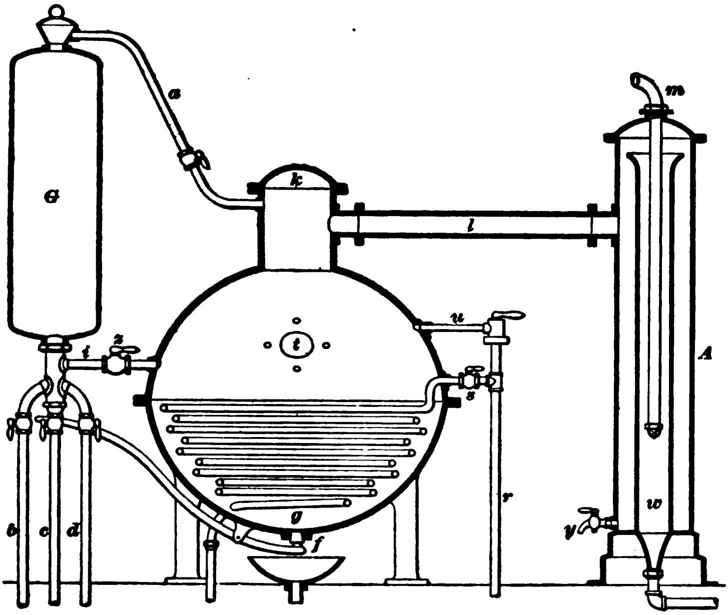

FIG. 9

47. Fig. 8 represents a perspective view and Fig. 9 a section of this form of evaporating pan. The boiling pan *B* consists of air-tight hemispheres surmounted by a funnel connected with a tube *l* that leads to a condenser *A*. The apparatus is supplied with steam by the pipes *r*, *s*, the steam circulating in the boiling pan by means of a pipe *g*. By opening a lever valve f, the juice can be run, by means of the pipe o, into the pan p, when the pan, after continually boiling, requires to be refilled. The pipes l and w are connected to an air pump. The manometer h shows the state of the air pressure, which can be regulated by opening the pipes connected to the vacuum chamber. By means of the gauge cylinder G, the quantity of sirup in the boiling pan can be ascertained, the gauge cylinder being connected to the boiling pan by the pipes a and i, and the height read off from the gauge tube n. For the purpose of ascertaining its consistency, the sirup can be removed from the gauge cylinder by means of either of the three pipes b, c, d. By pipe u, steam can be admitted to the boiling pan and condenser; e is a sight glass through which the state of the juice can be observed; q is a grease cock, water or paraffin being generally used to prevent the adhesion of the scum to the working parts of the pan. The condenser consists of the jacket A, arranged to prevent the mixing of the juice with the water used for condensation; x is a gauge; the pipe m conveying water to the condenser terminates in a rose.

48. The construction of the vacuum apparatus has lately been much improved. In the apparatus of Wellner & Jellinek, with horizontal heating pipe (Figs. 10 and 11), the bottom is formed by two sides converging obliquely. In order to shut the vacuum valves c at pleasure, they should be connected with the arrangement shown in the figure. This consists of an angle lever f, turning on the bolt f_3. With one of its arms f_1, the valve c is connected in such a manner that it can turn on d, while the second arm f_2 leans against the lower end of a projection of the sledge g. The sledge slides in a small tail-shaped groove h of the guiding piece i, and can be raised or lowered by turning the screw j. If the screw is raised, the valve opens in consequence of its weight and the pressure of the liquid, so that the contents can flow out. By screwing down g, the lever f is turned and the valve is shut. The figure represents for every valve c a special screw j, with its accompanying sliding piece g. But

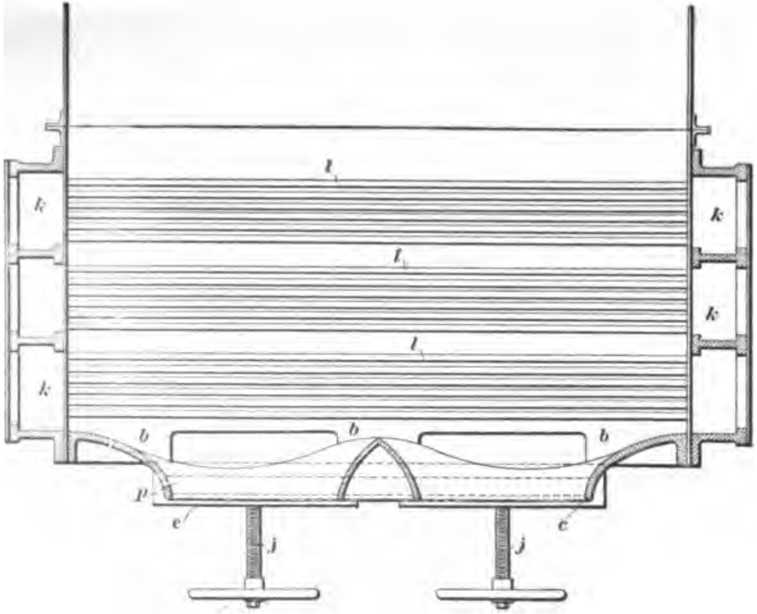
l
k
k
l1
k
k
l
k
b
b
b
p
e
e
j
j

FIG. 10

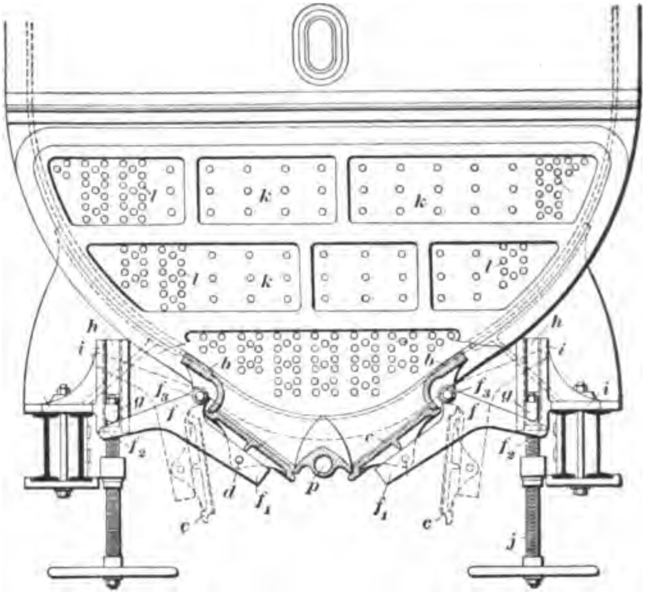
l
k
k
l
k
l
h
h
i
i
i
i
b
b
f3
f3
g
g
f
f
c
c
f2
f2
d
f1
f1
p
e
e
j

FIG. 11

the arrangement can be so made that instead of the bolt f_2, a shaft is introduced having as many arms f_1 as there are valves, and which can be turned by a single arm f_2. The heating steam circulates from the boxes k, through the horizontal tubes l. There is also a longitudinal channel in the bottom capable of being heated.

49. Working of the Vacuum Pan.—The general practice of working a single vacuum pan may be described as follows:

The air pump is started, and after a vacuum in the pan of 26 or 27 inches has been reached, the feed cock (on the side of the pan) is opened and a sufficient quantity of liquor is drawn in so as to completely cover the first steam coil. Steam is now turned in and the liquid rapidly concentrates. Fresh supplies of sirup are admitted at short intervals, the feed cock being opened for about 15 seconds at a time until the mass in the pan begins to show grain. The grain is fed carefully, the cock being opened frequently, and each time the quantity admitted is slightly increased. As the amount of the sugar in the pan continues to increase, steam is turned into the second and third coils, until, at the completion of the charge, the pan is filled to just below the sight glass. In this way, the grain "grows" in size. On the conclusion of the boiling the vacuum is destroyed, and the charge is run out into a tank and allowed to rest from 1 to 2 hours, when a further crystallization takes place.

It is customary to draw in as much sirup as will cover the bottom coil (when reduced by concentration), which is called *graining low down*. Some prefer to grain higher, while others prefer to grain when the pan is half full—*graining high*. An objection to graining high is that the grain has not so much time to grow, although this objection does not hold good in all cases. While it requires 7 hours to boil a strike of 8 tons of *masse cuite* graining low, not more than 6 hours will be required if grained high. The crystals in the second case will not be as large as in the first, although in an 8-ton pan they will be of a fair medium size, even by the quicker method.

50. The drawing is conducted as follows: The charging cock is opened and shut off again as soon as the liquid boils up to the bull's eye on the opposite side; the contents are quickly boiled down and the cock is opened again and shut off as before, when the liquor boils to the same height. This is kept up until all the sirup intended to form grain has been taken in; for instance, approximately 2,000 gallons of sirup, having a specific gravity of 18° to 20° Baumé, to a 5-ton pan would be the correct amount.

The granulating in the pan is readily recognized by the practical sugar boiler. Among the empirical tests may be mentioned the *thread test*. A drop of the liquid is taken up between the thumb and the forefinger, and the concentration is judged by the length to which a thread can be drawn out and the manner in which it breaks. It may be said, as a rule, that if it is possible to draw a thread about ¾ inch without breaking, the granulation point has been reached.

In boiling for large grain, it is essential to grain low. The grain commences to form minute specks, and these rapidly increase in size as well as in number, until the whole mass of liquor is filled with them. As each lot of sirup is admitted, it deposits on the grains already formed, causing these to grow larger and larger. During granulation, care should be taken not to allow the temperature to rise above 71° to 78° C. (160° to 173° F.), although this temperature may be raised later on, in order to harden the crystals. But this must not be done too soon after graining, or the contrary of the desired effect will take place; that is, the crystals will melt again.

51. Rules for Graining Sirup in the Vacuum Pan. The thinner the sirup that is admitted into the pan, the larger will be the crystals produced. For this large-grained sugar a few, but heavy, charges should be admitted, so as to give the grain enough time to grow; the larger the crystals required, the more quietly and slowly should the boiling be conducted. To make regular grain, granulation is brought about very slowly, and on no account must the grain be forced by boiling very high before the first charge.

In pan boiling, it is important to avoid forming "false grain." The two stages when this is most liable to happen are: (1) the time when the sulphuric acid (for producing yellow crystals) is admitted into the pan, and (2) the "opening" of the sugar when restarting the pan to "double," that is, when having struck out half the contents of the pan, fresh portions of sirup are admitted on to the *masse cuite* left in the pan. If the contents are not sufficiently high when sulphurous acid is admitted, false grains form while working up for striking. Unless the *masse cuite* be opened very slowly, the new lot of sirup, instead of depositing on the already formed crystals, and thus increasing their size, will form an independent grain, called *false grain*, which not only spoils the sugar but prevents the separation of the grain and molasses in the centrifugals.

When false grain appears predominant, the only remedy is to strike it out at once and spin it in the centrifugal, mixing it with warm water if necessary. When not very bad and the pan is not much more than half full, the heat and the addition of a few heavy charges of new sirup, as a rule, will remove it.

52. Demarara Yellow Crystals.—Sulphuric acid possesses the property to impart to the sugar the delicate yellow bloom so much admired in *Demarara crystals*. If too little is mixed with the *masse cuite* in the pan, the original color of greenish gray is very little, if at all, improved; if too freely administered, the sugar will turn red in a day or two after curing. It is admitted, last of all, that it is not permissible under any circumstances to make a charge of sirup into it immediately previous to striking. The quantity of sulphuric acid required depends principally on the color of the *masse cuite*. As a rule, 3 gallons of sulphuric acid diluted with 1½ gallons of water, added to 5 tons of sugar, is about the right proportion. In all cases, the least quantity that will produce the desired effect should be used.

53. Of great importance is the proper striking point; it arrives when a sample taken by means of a proof stick will hardly run out of the socket of the same. *Masse cuite*, on leaving the pan, should have a light-red golden color and a temperature not exceeding 60° C. (140° F.). The object of doubling is to increase the size of the grain, thus enhancing the market value of the product and saving time. Some sirup makes sugar that will stand doubling from 2 to 5 times, while other sirup will get sticky after the first cut of the pan. It is essential to take great care while opening the *masse cuite* left in the pan; for the third and fourth cut a temperature of 74° C. (165° F.) may be maintained while opening slowly and carefully, the operation requiring 15 to 20 minutes. The drawing in of sirup demands more care in consequent cuts than in the first.

A considerable loss of sugar is caused by doubling, depending on the amount of acid used and on the quality of the sirup. The same is estimated to amount to 20 to 25 per cent. of the sugar, and some believe that a better return is obtained from the larger quantity of dark sugar at lower prices. But, on the other hand, the loss really means sugar converted into a high grade of sirup of a beautiful golden color, and the extra market value of the yellow crystals is said by some of the best authorities to more than make up for the extra cost and increased inversion of crystallizable sugar.

When sour canes are accidentally sent to the boiler house, the sugar is apt to get sticky in the pan, and occasionally to such a degree as to interfere with the formation of grain, thus endangering the whole strike of sugar. In cases where the stickiness is only moderate, two to three buckets of strong lime water, taken into the pan through the acid cock, will remove the trouble. Besides this, the excess of acidity should always be neutralized by lime water, leaving the sirup only slightly acid before drawing into the pan.

54. Molasses.—*First molasses* runs from *masse cuite* that has no molasses boiled in it, *second molasses* drains from *masse cuite* that has been boiled together with molasses, and *third molasses* drains from vacuum molasses sugar. These are kept distinct. Third molasses is so sticky and impure that it can only be utilized in the manufacture of rum; the same is also sometimes the case with second molasses, if the market price of sugar does not warrant its conversion into sugar. Only first molasses should be used for mixing with sirup sugar in the pan, and second molasses for boiling molasses sugar (third sugar); second molasses should never be used for boiling with pure sirup sugar in place of first molasses. There is a great difference of opinion about the boiling of molasses, but the following plan is one of the most usual, provided the general arrangements of a factory permit the molasses to be boiled within an hour or two after separation in the centrifugals.

Assuming that the pan has struck out 3 tons, being refilled and cut a second time, leaving it still half full, for a third time fresh molasses, tempered with lime water and reduced with water to a density of 30° Baumé, is drawn in. The contents struck out and "spun" in the centrifugal should yield 2½ to 3 tons of second sugar; that is, sirup sugar with which molasses has been boiled, giving about 1.2 tons of sugar from molasses, much improved in color, in addition to the 2 tons obtained from the sirup, and upon which the molasses was admitted. To make a very pale sugar, however, this process will not answer, and the molasses must be utilized by being made into a fine quality of third sugar, or, better, into rum.

55. For tempering molasses, lime water should be stirred in until the point of neutrality has been nearly reached. For second and third sirups, or molasses that is to be boiled for grain, the density must be reduced to 30° Baumé, either by blowing in live steam or, if this is not convenient, by the addition of water. The boiling is performed in an exactly similar way to first sirup, except that no attempt should be made to try for large grain, as the impurities present effectually prevent the grain from growing beyond a certain limit. It is not an unusual custom to considerably increase the temperature before striking, by dropping the vacuum 2 or 3 inches, which is readily done by checking the supply of water to the condenser and keeping the steam full on the coils and jackets. The temperature of the *masse cuite* is then about 77° C. (170° F.), whereas it has previously been about 60° to 74° C. (140° to 165° F.). The object of this is to procure a hard grain, which may be washed into the centrifugal. The *masse cuite* from second and third sirups should always be allowed to stand 2 or 3 days in the coolers, to grow crystals, before being placed in the centrifugal. Molasses from third sugar of a density of 34° to 36° Baumé is always "jellied," or "boiled smooth," and it is then not necessary to reduce the density. If very acid, however, it should be tempered to neutrality and boiled until a sample will draw out to a thread from 1 to 1½ inches in length between the fourth finger and thumb. At this stage and before any sign of granulation has commenced, the contents of the pan is discharged into a cooler and allowed to stand for a period of not less than 1, and not exceeding 2, weeks, after which time they may be passed into the centrifugal.

CURING THE SUGAR

56. It is easily understood that the products of the various operations described in the preceding articles differ widely in character and nature and demand separate treatment in their preparation for the market. This treatment, or whichever treatment is employed, is known as *curing*, and embraces the whitening or bleaching of the sugar. The several methods that may be employed will be discussed in succession.

57. Simple Drainage.—The oldest method of curing the sugar consists in drainage, and the product thus obtained is crude and unsightly. In order to remove a certain amount of molasses and other impurities and to improve the quality and appearance of the sugar, the semiliquid mass is removed from the coolers as soon as the same is sufficiently cooled and placed in caskets with perforated bottoms, the perforations having been loosely filled with twisted leaves or rushes (the latter sufficiently long to reach above the contents of the casket) in such a manner as to form the roughest imaginable strainer. The caskets are placed on a scaffolding over a large tank. In this way the draining process slowly and imperfectly goes on, a portion of the molasses escaping into the tank below, but a considerable part still remaining with the sugar. In fact, the separation of the molasses and sugar is so imperfect and incomplete that a considerable waste and leakage occurs when the sugar is transported to the refineries. Sugar cured in this way is known as *muscovado sugar*, and is the most impure form of raw (*grocery*, *moist*, or *brown*) sugar. It is now produced only on a very small scale in some backward countries.

58. Claying.—The first improvement of this oldest and crudest method was introduced by the Spanish and Portuguese on the sugar estates in Cuba. It is based on the fact that the impurities present in muscovado sugar are much more soluble in water than is the sugar by itself. Thus, a judicious washing with water will effect a considerable purification. The original method of washing consists in placing the sugar in inverted cones with very small openings in their apexes, which are stopped up during the filling and for 12 hours afterwards. Upon the mass of sugar in the cone a batter of clay and water is placed—hence, the name *claying*—the object of this being to insure a very gradual percolation of the water through the mass. This water, on its downward course, carries with it the uncrystallizable sugar and coloring matter embedded between the crystals of the sugar. The resulting sugar is considerably lighter than muscovado, but its grain is rather soft, and, as will be readily understood, the whole operation is far from being economical.

59. Washing With Alcohol.—The very slight solubility of sugar in alcohol and the ready solubility of its impurities in this medium suggested the practice called *spirit washing*. This process simply consists in substituting alcohol, or a mixture of alcohol and water, for the water used in claying. The results are, however, not in accordance with the cost of the alcohol and the market of the sugar, and so this method, which was chiefly employed in the East Indies, has been nearly entirely abandoned.

60. Vacuum Chest.—The vacuum chest consists of an iron box with a tray of wire gauze above and connected with an air-pump suction below. The sugar is spread on the tray, and the downward suction produced by the working of the air pump creates a tendency in the liquid portion of the mass to separate itself from the solids. Effectual separation, however, can only be obtained when the crystals of the sugar are comparatively large and hard, while with soft and small crystals the process proved unsuccessful.

61. Centrifugals.—All the methods just described have been practically superseded by the centrifugal or hydro-extractor. There are a large number of different makes on the market, but all consist essentially of a drum revolving on a vertical shaft, the sides of which are formed of wire gauze or perforated metal for holding the sugar. The drum is surrounded by a casing for a distance of about 4 inches, the annular space thus left being for the reception of molasses, which is expelled by the centrifugal force through the sides of the drum when the latter revolves at a high speed. A spout conducts the molasses from the annular space to a receiver.

62. There have been a great many improvements in the processes described during the past few years, which have resulted in economizing the cost of production to a considerable extent. These improvements have been introduced in the sugar-cane districts of this country, and are similar to the processes used in the manufacture of beet sugar, which is fully described in *Manufacture of Sugar*, Part 2.

MANUFACTURE OF SUGAR

(PART 2)

BEET SUGAR

INTRODUCTORY

1. The manufacture of beet sugar has been practiced in Europe for about one hundred years, and nearly all the sugar consumed there is obtained from this source. The beet-sugar industry in this country, however, is of very recent date, but through the efforts of Doctor Wiley, Chief Chemist of the United States Agricultural Department, E. H. Dyer, Henry D. Oxnard, Claus Spreckels, and others, this industry has been growing wonderfully and promises to take immense proportions.

2. Beets thrive best in a temperate climate, which, in the United States, covers an immense area. While the plant develops under a great variety of weather conditions, more recent experience seems to confirm in a measure the previously accepted theory that the sugar beet, as a rule, does best in regions where the mean temperature for the months of June, July, and August averages 70° F.

Sunshine is required to make sugar; hence, the number of clear and sunshiny days that can usually be depended on in any section is an important consideration. This explains

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the advantage of many parts of the so-called arid West for this industry, especially California and the Southwest. Another climatic consideration is favorable weather during the ripening and harvesting period of the beet. Clear sunshine and absence of fogs and moisture are also important at this period.

3. Variety of Beets.—All kinds of sugar beets are practically identical with the common garden beet, *Beta vulgaris.* Different varieties have arisen by reason of special selection and culture producing some valuable peculiarity in the beet. These accidental valuable qualities, by careful selection, have become fixed, and are associated with certain external properties, which have thus come to be regarded as distinguishing characteristics. The shape and size of the beet, its color, the character of its foliage, whether erect or spreading, are the most usual marks of distinction. The beets are also most frequently designated by the names of those who have developed them, by the name of the locality in Europe in which they were grown, or sometimes by their color. Among the most frequently occurring varieties in Europe may be mentioned the Vilmorin Improved, Klein Wanzlebener, Improved Klein Wanzlebener, White Excelsior, White Imperial, Rose Imperial, White Silesian, etc. The varieties most popular in the United States, especially in California, are the Vilmorin and the Klein Wanzlebener, while Nebraska growers prefer Dippe la plus Riche, Klein Wanzlebener, Original Klein Wanzlebener, and Vilmorin Improved White; the latter two varieties are also chiefly grown in Utah. It should be borne in mind, however, that no variety of beet is suitable to all conditions and climates. Different soils and treatment make peculiar demands on the variety, and the experience with varieties in other parts of this or foreign countries is not a safe guide.

4. An ideal beet as to shape and foliage is shown in Fig. 1. It is assumed that beets weighing more than

3 pounds are, as a rule, of inferior quality, but this depends to a greater or lesser extent on the variety of seed, the suitability of the soil, and the conscientiousness with which the latter has been prepared and cultivated. As a general rule, however, it must be said that the larger beets are lower in quality, as far as the sugar content is concerned, than the smaller or medium-sized ones. The beets most eagerly sought by sugar manufacturers are those that weigh between 1 and 2 pounds. Where beets are too large or too poor in quality to be used in sugar manufacturing, they must not be considered entirely valueless, as they may be well and advantageously utilized as feed for live stock.

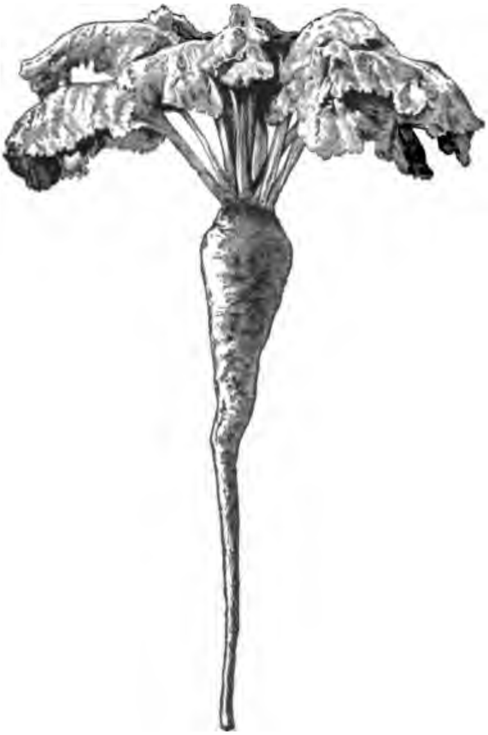

FIG. 1

Fig. 2 represents a cross-section of a beet of natural size at its largest diameter, the dotted lines showing the concentric rings of growth.

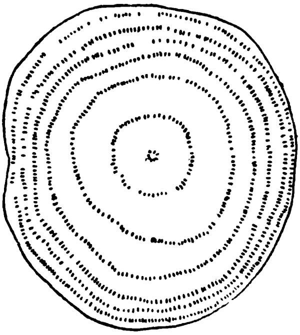

FIG. 2

5. Planting.—The quantity of seed required per acre varies greatly. The seed germinates more quickly in some soils than in others, and many things unite to act against the germination of every seed, so that it has been found best to plant enough to insure a good stand, and also to exercise the force necessary to lift the crust of the soil and allow the tender sprouts to reach light and air. It is generally conceded that each acre requires from 12 to 20 pounds of seed. The rows should not be less than 18, nor more than 20, inches apart, this space being sufficient for a horse to walk in during cultivation and permitting the leaves of the full-grown plant to shade the ground. The proper depth for planting depends very much on the locality; in moist ground and a humid atmosphere, ½ to 1 inch is fully deep enough to lay the seed, while in very arid regions, 1½ inches is necessary, owing to the rapid evaporation of the surface moisture. Where conditions permit, shallow planting is certainly advantageous.

6. Cultivation.—When the seed has sprouted about 2 inches above the ground, it will be found that the plants stand too close to one another and it becomes necessary to thin them so that the remaining ones stand a uniform distance apart. This is done most economically by using a hoe 4 inches wide and having a sharp point. Spaces are then struck out with the hoe and small bunches of beets, 6 to 8 inches apart, are left for two or three "thinners," who deftly pull out all but one beet, leaving the healthiest. More than one beet in a place means ruin; in the struggle for supremacy, they become twisted around one another, and at the end none of them show a healthy growth or are really worth harvesting. Therefore, it should be a very strict rule never to leave more than one beet in a place. In poor soil the beets should be thinned until about 10 inches apart; in fairly rich soil, 8 inches is preferable; and in very rich soil, the distance can be shortened to 6 inches. Large plants, as has been stated, are not the most desirable. After thinning, the plant, as a rule, will look weak and sickly, and it generally requires about 48 hours before it recovers sufficient strength to again hold up its head.

7. The sugar beet is a plant that requires plenty of nourishment, and it is to the farmer's interest to keep the soil as clear as possible from weeds. Cultivation should begin soon after the thinning has been accomplished and should be frequently repeated. The old-fashioned ground cultivator is not suitable for this purpose, and numerous beet cultivators have been constructed, the Moline beet cultivator being considered the most efficient, and is largely used at present. Whatever cultivator is used, the rule is, "keep the soil mellow, the weeds down, and nature will do the rest." The crown of the beet, from where the foliage sprouts, should not be covered by the soil, nor should the beet be exposed too much. The laceration of the leaves should be avoided after they are large enough to protect the exposed part of the beet from the heat of the sun, and from this period until the time of harvesting arrives, it is desirable to keep the field free from weeds by hand, one man being able to keep a field of 20 acres clean.

8. Harvesting.—The exact time of maturity of the beets can only be determined by chemical analysis, but experience has demonstrated that when the period of maturity arrives the dark-green color of the leaves disappears, the outer leaves wither and die, and the center leaves assume a reddish golden-yellow color. Beets seeded very late in the season often fail to show these characteristic marks of maturity, yet the analysis shows a high percentage of sugar with a corresponding high coefficient of purity. When the chemist discovers that the beet is at its highest sugar content and coefficient of purity, say from 14 to 20 per cent. sugar, with a coefficient of from 80 to 85, the crop should be harvested as soon as possible.

9. Beet Plows.—The most necessary article in harvesting the beet, next to wide-tired and strongly built wagons, is the beet plow; it is poor economy to try to plow out the beet crop with an ordinary plow. The loss in roots broken off in the ground and those missed entirely will more than pay for a suitable plow in harvesting any considerable acreage. The ordinary subsoiler supplied by the trade, of which the beet plow is a type, is not strong enough to stand the test. A very convenient plow, which loosens the beets in the ground and at the same time tops them, is shown in Fig. 3. The machine is followed by children, who remove the loosened beets from the ground. Where topping is not performed by machinery, a strong, well-riveted butcher knife, with a 10-inch blade, is fully as useful in performing the work as a costly knife made expressly for the purpose.

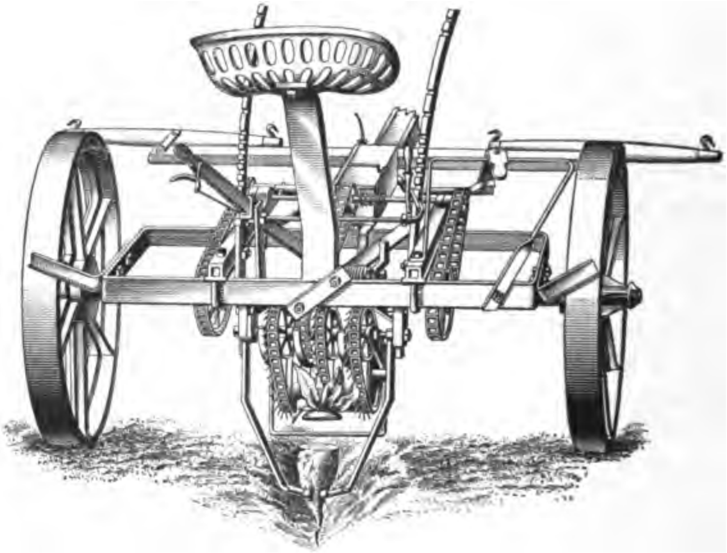

Fig. 3

The plow lifts the beet root partially out of the ground, so that it can be easily grasped by the left hand of the topper, while with the right hand he deftly cuts the tops off at one blow, cutting just below the lowest leaf growth.

10. Beds for the Topped Beets. — The plowman is perhaps a hundred rows ahead of the toppers, and must precede them with a rake; at frequent intervals he removes the beets a little, making a bed about 6 feet across with a smooth surface. If the crop is good, these beds should be about 30 feet apart and should be in perfectly straight rows. The reason for this is readily seen. The topper throws the finished beets to the bed, where they are ready in heaps for the loader, unmixed with leaves or dirt. The leaves are very objectionable to the factory, as they clog the knife in the slicer and in other ways hinder the operation. The clods of dirt and the leaves cause extra work in determining the net weight of the farmer's load, so it should be the aim of the farmer to have the beets in clean heaps, as this will facilitate all interests involved. The determined solids or salts absorbed by the beet in its growth centralize in the top of the beet, particularly in that portion grown above the surface. If the topping is properly done, all that portion grown above the ground is cut off square across, then the analysis of mature beets will ordinarily show a satisfactory coefficient of purity, which should be always above 80 per cent.

11. The content of sugar in different parts of the beets is shown in Fig. 4. Although the beet tops contain less sugar and are richer in mineral salts than any other part of the beet, they are not without considerable value. While the practice of using them as a stock feed should be discouraged, their value as a fertilizer cannot be overestimated, and the best way to dispose of them is to plow them under as soon as the field is cleared of the beets.

The following is an extract from a French work, "Traite de la Culture de la Betterave à Sucré," by G. Dureau, showing that the value of beet-sugar tops as fertilizer is well appreciated in the older beet-raising countries:

"On a well-conducted farm, the beet tops and leaves should be well looked after; we have shown that the leaves and tops of rich sugar beets equal 50 per cent. of the weight of the roots. If we take, for example, a yield of 50,000 kilos of beets to the hectare (22$\frac{6}{10}$ tons to the acre), with, say, 25,000 kilos (11$\frac{3}{10}$ tons per acre) of tops and leaves, the latter will contain 95 kilos of nitrates, 63.75 kilos of phosphoric acid, and 270 kilos of potash (or, say, 85 pounds of nitrates, 57 pounds of phosphoric acid, and 241 pounds of potash to the acre).

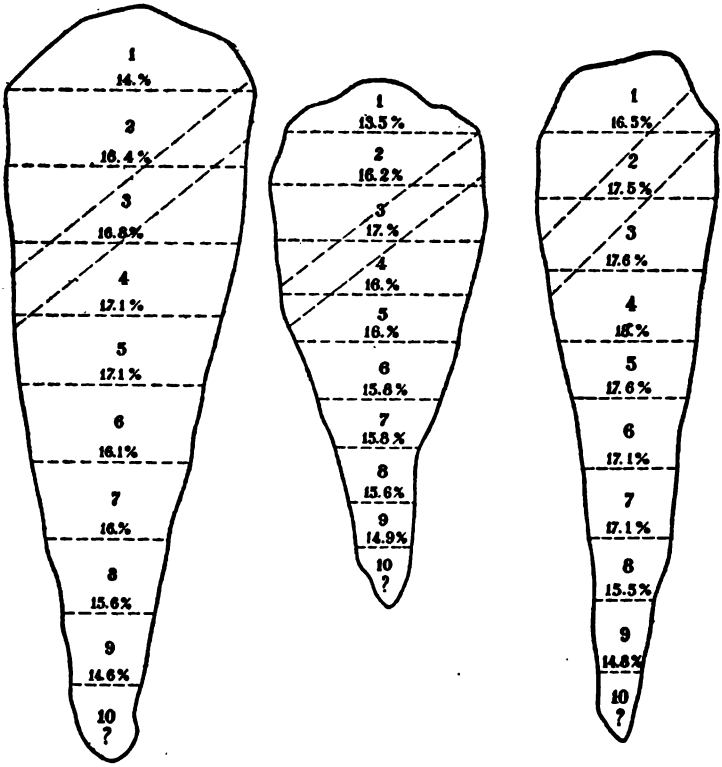
1
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FIG. 4

"On account of their richness in nitrates, phosphoric acid, potash, and more or less useful elements, beet tops are an excellent fertilizer, and in every instance should be restored to the field that produced them, by plowing them under while green. Different experiments have proved that it is better to plow them under green than dry.

"If the beet tops are not restored, the value of the elements lost to the land may be estimated as follows: Phosphoric acid at .5 franc, potash at .25 franc, nitrate at 1.5 francs per kilo, or, say, a loss of 241 francs per hectare for a yield of 50,000 kilos of beets ($\frac{19}{50}$ per acre for a yield of $22\frac{6}{10}$ tons). It is, therefore, a serious mistake to underestimate the benefit of the beet tops as a fertilizer; they are of great value, and every farmer that restores them to the soil realizes a double profit in the chemical and physical improvements of his land."

12. Storing the Beets.—After the beets have been harvested, it becomes necessary to store them, as each factory has only a limited daily capacity. In the mild climate of California, it is possible to keep the beets without injury to them in large sheds without any further precautions. In colder climates, a different way of storing has to be selected, especially as it is the rule with most sugar factories that the grower has to do his own storing until called on to deliver the beets. A method of storage should be chosen that retards the evaporation of the water in the beet, as the beet that has lost 10 per cent. of its water is, as a rule, not suitable for the manufacture of sugar any longer, and, besides this, the beet must be protected from both overheating and freezing, as these tend to produce decomposition. *Siloing* is the mode of storage that has been used in European countries effectively for many years, and this method has also found favor in this country, being convenient and economical.

For the purpose of making a **silo**, a few rows of beets are dug out and a tongue scraper is run over the place, making a shallow trench. As the beets are dug and topped, they are thrown into this trench and covered with leaves, a furrow is plowed down each side, to drain off the water in case of storms, and the leaves are covered with soil, to keep them from blowing away. As the season of cold weather approaches, this cover of soil should be increased according to the climate; the cover varies from 20 to 75 centimeters. Care should be taken in siloing beets not to throw into the silo those that have been injured in any way, as they are very liable to decompose and infect the other beets.

13. Chemical Constituents of the Beets.—The flesh of the beets consists of a quantity of small cells containing a clear, colorless fluid. An analysis of the flesh of the beets shows the following constituents:

Water	82.6%
Sugar	11.3%
Cellulose	.8%
Albumen, caseine, and other bodies	1.5%
Fatty matter	.1%
Organic substances: citric-acid pectin and pectic acid, asparagin and asparic acid, and betain.	
Organic salts: calcium potassium and sodium, oxalate and pectate.	
Inorganic salts: potassium nitrate and sulphate, calcium and magnesium phosphates	3.7%
Total	100.0%

The components of a beet vary according to the time of the year. At some periods it contains more water than at others, from 82 to 84 per cent. being about the average. In the autumn it does not contain slime sugar; in February and March, the components intermingle, and some decrease nearly 2 per cent., as is shown from the following analysis:

	OCT.	FEB.
Woody fiber and pectin	3.49%	2.52%
Water	82.06%	84.36%
Sugar	12.40%	10.60%
Slime sugar	.00%	.65%
Mineral salts	.75%	.63%
Organic acids and extractives	1.30%	1.24%

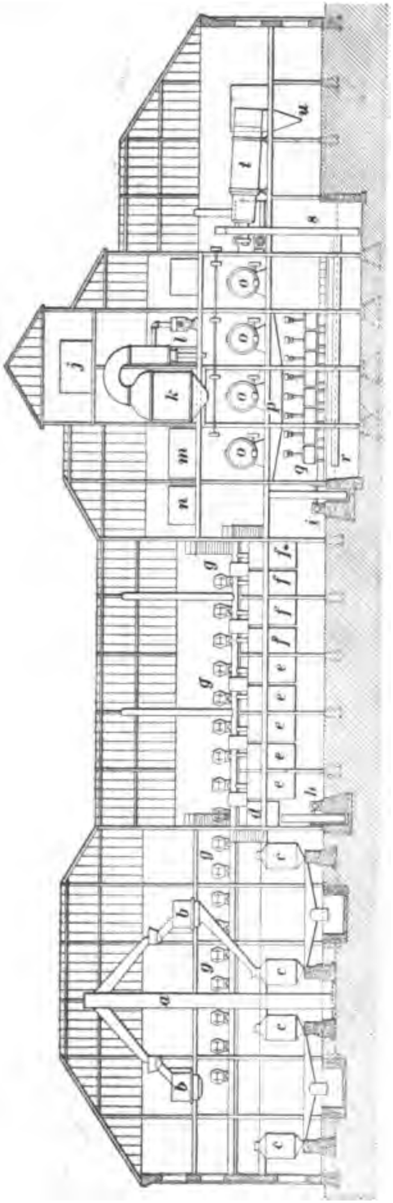

FIG. 5

14. Arrangement of the Modern Sugar Factory.—Fig. 5 represents a plan of the Los Alamitos sugar factory, near Los Angeles, erected in 1897, by E. H. Dyer and Company. Referring to this illustration, *a* represents the beet elevator; *b*, the beet cutter from which the sliced beets, called *cossettes*, are delivered into the diffusion battery *c*; *d*, a receiving tank; *e*, the first carbonators; *f*, the second carbonators; *g*, the filter presses; *h*, the engine that drives the beet department; *i*, the engine that drives the sugar department; *j*, the tank to supply the diffusion battery; *k*, the strike pan; *l*, the air pump, to remove air and gases from the strike pan; *m*, the tank to hold the concentrated juices preparatory to being worked in the strike pan; *n*, the water tank; *o*, the crystallizers; *p*, the mixer; *q*, the

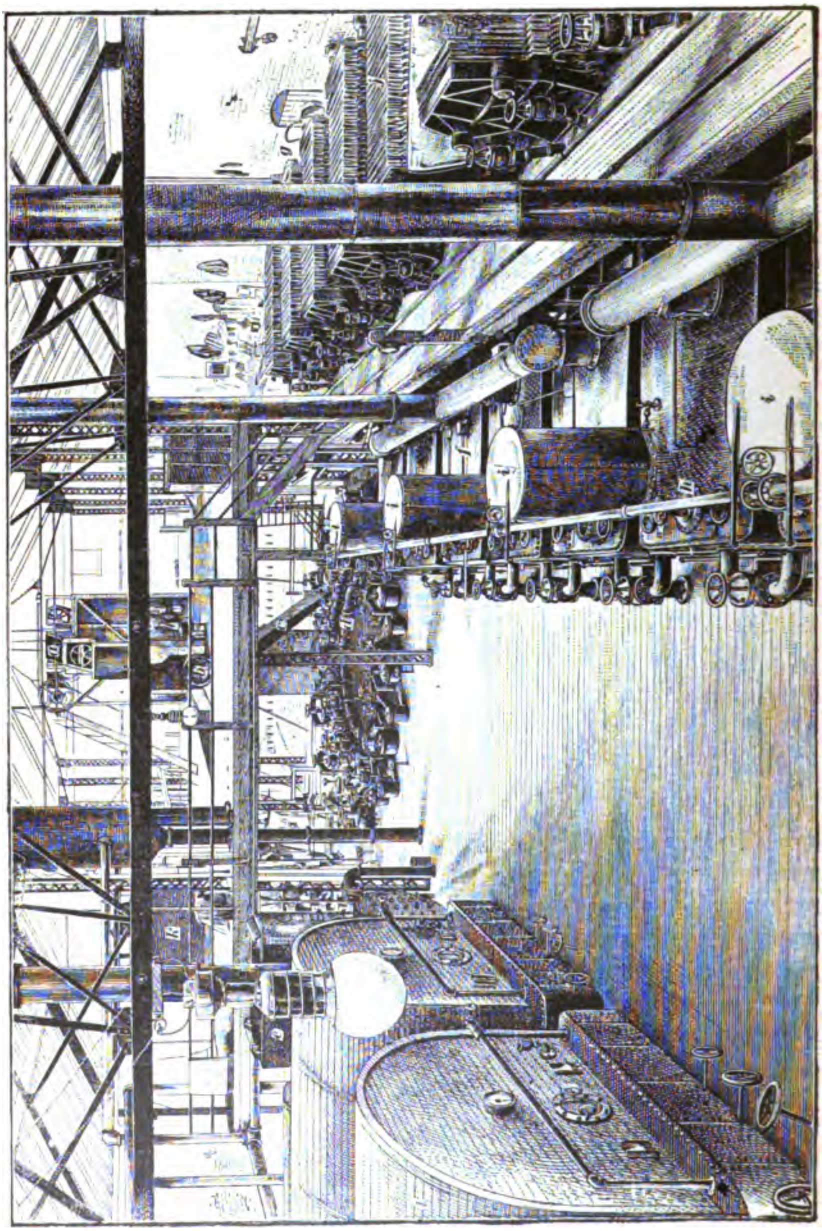

FIG. 6

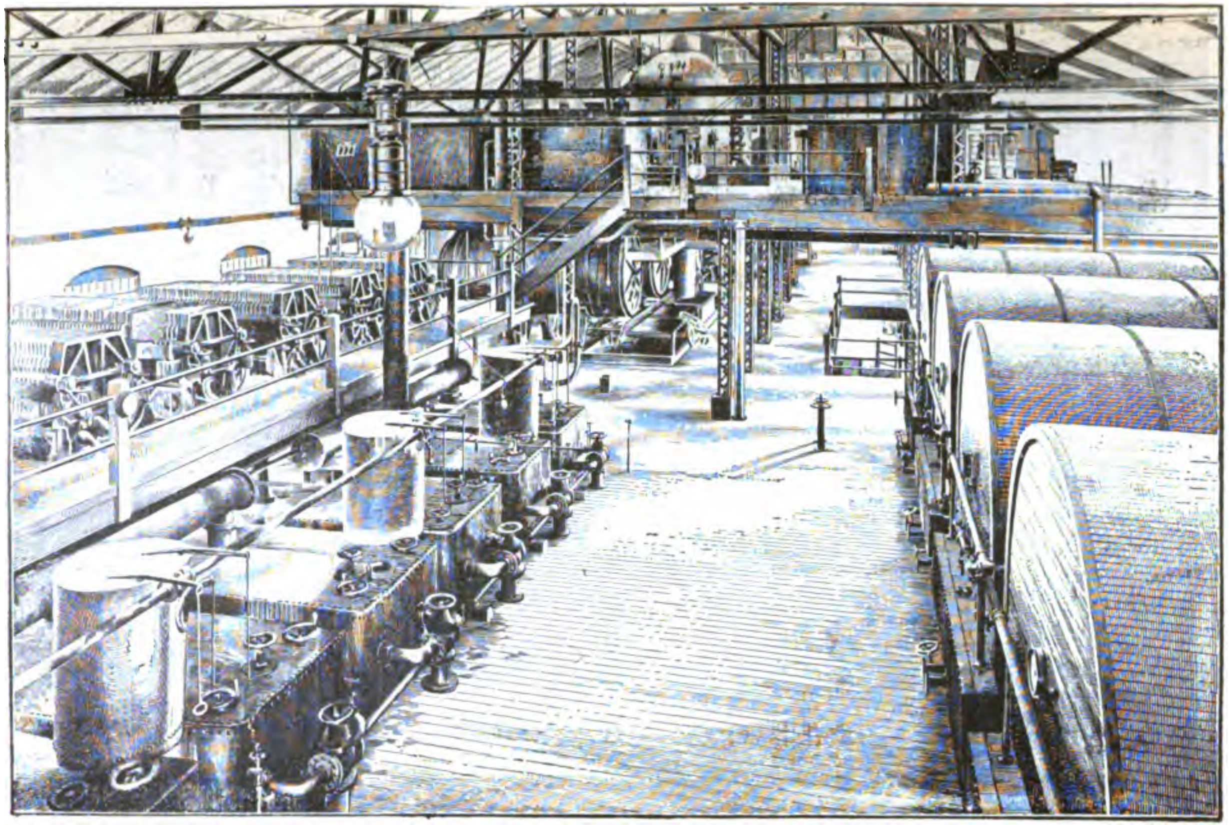

FIG. 7

centrifugals; *r*, the conveyer that carries the sugar from the centrifugals and delivers it into the elevator *s*; *t*, the sugar drier; *u*, the barrel packers.

15. Figs. 6 and 7 will give a very clear idea of the main floor of a modern sugar house, *a* being the elevator that raises the beets from the washer and drops them into the cutter *b*, Fig. 6, where they are sliced. These slices are then conveyed through the chute *c* into the cells of the battery *d*. The exhausted cossettes, or pulp, are dropped below the battery and carried outside, where they are loaded on cars or wagons, to be taken away for feed for cattle or stored in silos for future use. The diffusion juice leaves the battery and is measured in the measuring tanks *e*. From there it goes to the first carbonators *f*, then to the first filter presses *g*, and then to the second carbonators *h*. The lime-measuring tanks, for supplying milk of lime to the carbonators, are shown at *i*. From the second carbonators, the juice goes through the second filter presses *j* and then to the sulphitation tanks *k*; after leaving there it goes through the mechanical filters, not shown, and thence to the quadruple-effect evaporators *l*. Now, being thick liquor, it is again run to the sulphitation tanks and the mechanical filters, and from there to the pan-supply tanks *m*, Fig. 7, and then to the vacuum pan *n*. From there it is dropped as magma or melada to the mixer *o*, and is taken into the centrifugal machines on the floor below, where the crystallized sugar is separated from the molasses, which, after being boiled, is dropped into the crystallizers *p*.

MANUFACTURE OF BEET SUGAR

GENERAL OUTLINES

16. The following description of beet-sugar making is partly taken from a pamphlet by William H. Holabird. The beets are delivered by wagon and weighed; the weighed beets are unloaded in storage bins with sloping bottoms, through which runs a stream of water confined in a flume with a movable covering. The beets are allowed to fall into this flume, the water floating them to the factory, where they fall into an elevator, which drains the water and carries them to a washer; here they are subjected, by means of propeller arms, to a thorough washing, after which they are automatically ejected into another elevator, which carries them to the third floor, where they fall into the slicer; here they are cut into long **V**-shaped strips about ⅜ inch wide by ⅛ inch thick and of various lengths. From the slicer the beets are transported by gravity through a chute to a diffusion battery, where the sugar is extracted by a series of leachings with hot water, the sugar being held in the solution in the cells of the beets, which cells also contain most of the impurities. The object of the diffusion process is to obtain the sugar with as few impurities as possible.

When sufficiently concentrated, the juice is drawn off by measure, enough being taken to extract the sugar without too great a dilution. This is accurately measured and a record kept of the time, number of cells, and density. This juice flows by gravity into a heater (calorisator), where it is heated to 190° F. This is important, as it is necessary to coagulate all the albuminoids before the presence of lime. From the heater the juice flows to the carbonator, where to the heated juice is admitted from 2 to 3 per cent. of lime in the form of milk of lime. This lime combines with the greater part of the impurities and forms an insoluble precipitate. The lime also combines with the sugar, forming calcium sucrate, which, if not decomposed, would be lost during filtration. This is done by injecting carbon dioxide, which is made in burning the lime used in clarification, forming an insoluble precipitate of calcium carbonate. Just enough gas is admitted to break up this combination of lime and sugar. Care is taken not to carry the operation too far, as after the calcium sucrate is destroyed the carbonic acid attacks the compounds of calcium and in time will liberate all impurities again. The process is very closely watched, and samples are taken every few seconds as the operation approaches completion. The samples are taken in a test tube. The proper point at which to stop the flow of the carbon dioxide is indicated by the formation of a granular precipitate showing clear liquor between the particles. The gas is instantly shut off, a test sample is sent to a table near by, where a chemist's assistant is stationed, and the percentage of the lime in the juice determined by titration with standard acid.

After clarification (or carbonation, as it is called in a beet-sugar house), the whole contents of the carbonator—juice and precipitate—are drawn off and forced through filter presses by means of a pump. The physical condition of the precipitate is important. If the operation has been carried on properly, the juice will filter rapidly through the filter presses, leaving a hard, porous lime cake that is easily washed and cleaned from the presses; but if improperly done, there will be a flocculent, gelatinous precipitate. The juice as well as the wash water from these filter presses is received in another set of carbonators, where about ½ per cent. of milk of lime is again added. This time there is not so much danger of overcarbonating with carbon dioxide, which is passed through the juice until there remains from .025 to .05 per cent. of lime in the juice. This is determined by actual test each time. The juice is then boiled, to precipitate the double carbonate that may be in the solution, and again forced through another set of filter presses. The clear, strained juice, which is now a light straw color, is pumped to the sulphitors, which are somewhat of the same construction as the carbonators. Here sulphur dioxide is forced through the juice by means of an air pump, which decolorizes the juice and precipitates the remainder of the lime. This juice, which is now water-white, is allowed to flow through mechanical filters, which eliminate whatever mechanical impurities remain in the juice. At this stage the juice, while comparatively pure, is dilute, containing only from 3 to 10 per cent. of sugar, depending on the quality of the beets being worked. It is desirable to concentrate it, which is done in a multiple-effect evaporator. The juice flowing through the effects becomes more condensed as it passes along, going in at a density of about 10° Baumé and coming out at about 30° Baumé, which not only concentrates the sugar, but also the impurities. This, which is technically called *thick liquor*, is again submitted to the fumes of sulphur dioxide, which neutralizes it, destroys the coloring matter, etc. The thick liquor is again passed through mechanical filters, which remove any foreign solid matters, after which it is boiled in the vacuum strike pan, where the sugar is crystallized.

When the operation is complete, the whole mass, known as *magma*, is concentrated until it contains in composition about 75 per cent. of sugar and 25 per cent. of molasses. The magma is then removed into a large iron tank with propeller arms constantly revolving, which is termed a *mixer*. Attached to and directly underneath the mixer are a number of centrifugals. After the magma has been in the revolving centrifugals a few minutes, it is entirely freed from the sirup; then a jet of water is sprayed on it, to remove the last trace of molasses, the machine is stopped, and the sugar falls through an opening in the bottom into a conveyer, which carries it to an elevator connected with a drier. Here the sugar remains in constant motion until every particle of moisture is removed from it; then the sugar passes from the drier over a screen, which removes any lumps that may have formed, and falls into a hopper, from which it is sacked or barreled and is ready for the market.

The molasses that comes from the magma is again boiled in the vacuum strike pan and another crop of sugar obtained from it and also another lot of molasses. This second lot of molasses is too poor in sugar to be crystallized in the vacuum pan; it is, however, concentrated to what is called *string proof* in the vacuum pan and dropped, when finished, into crystallizers, where it is continually agitated, which, with careful attention to temperature, causes all sugar that can be obtained to crystallize.

DETAILED DESCRIPTION OF PROCESS

17. When the beets are delivered at the factory, after having been deprived of their leaves, rootlets, and necks (the portion growing above the ground), they are received by an overseer, who weighs them and roughly estimates the *tare*, which is to be deducted for earth, badly trimmed necks, and other useless matter. As most of the beets received in the factory have been topped with more or less care, and have, as a rule, variable quantities of earth and pebbles adhering to them, it has become a rule to make a more accurate determination of the tare. As nearly an average sample of the beets as is practicable is selected. This sample should consist of as many beets as can be conveniently taken, the larger the number the better. This number may afterwards be reduced by the method of quartering. The roots are weighed, then thoroughly washed and dried by exposing them to a free circulation of air for a short time. The beets are again weighed, and the difference between this weight and the first is considered the tare.

18. Washing the Beets.—Fig. 8 shows the plan, side, and end elevations of a working drawing of one of Dyer and Company's beet washers. The washer consists of a shell, within which is a hollow metal shaft *b*, carrying the arms *c*, which agitate the beets in the water. The beets conveyed from the beds and sheds through a flume containing running water are raised by means of an elevator wheel and thrown upon an apron, which drops them into the washer having a driving gear *d* and *e* at its end. The beets are thrown out of the washer at the opposite side by the arms *f* on to a small conveyer, or into what is called a *trash catcher* (a sheet-metal box with a screw for conveying the beets from the washer to the elevator), which delivers them to the automatic scales that registers their weight and drops them directly into the cutter. Referring again to Fig. 8, *g*, *h*, and *i* are doors in the end and sides for cleaning out the dirt, sand, stones, etc., which are separated from the beets and

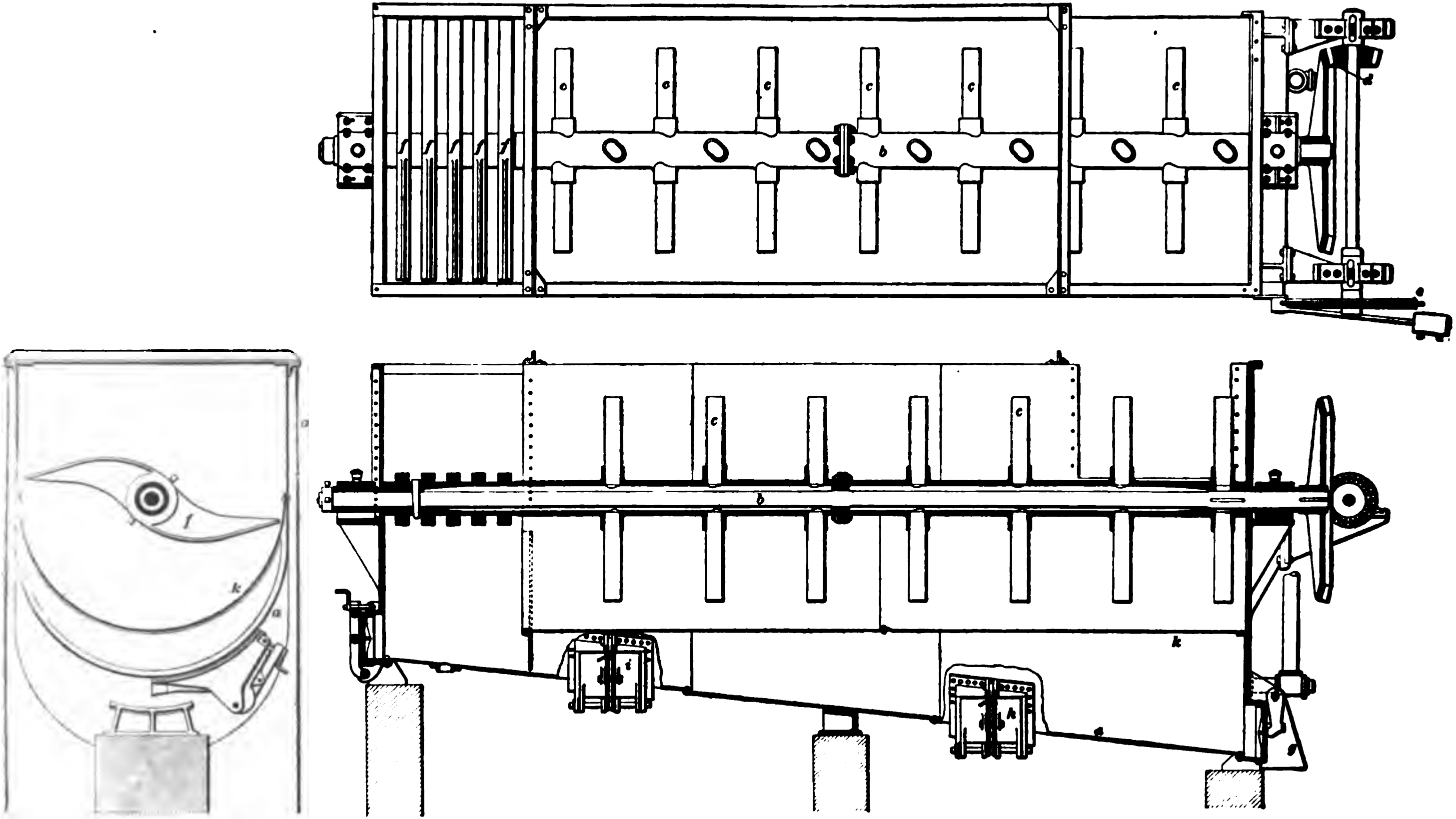
c
b
d
e
f
a
k
i
h
g

FIG. 8

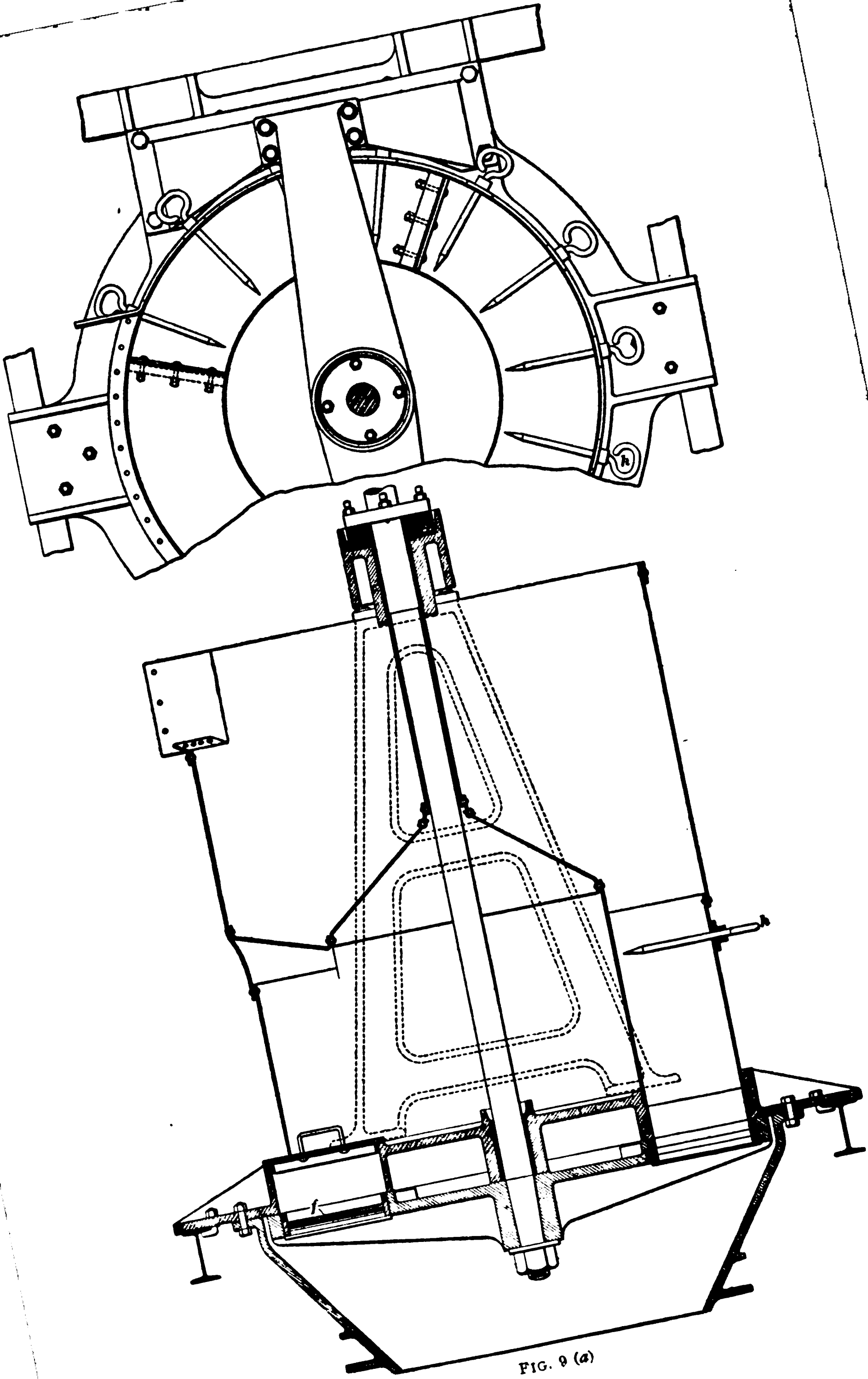

FIG. 9 (a)

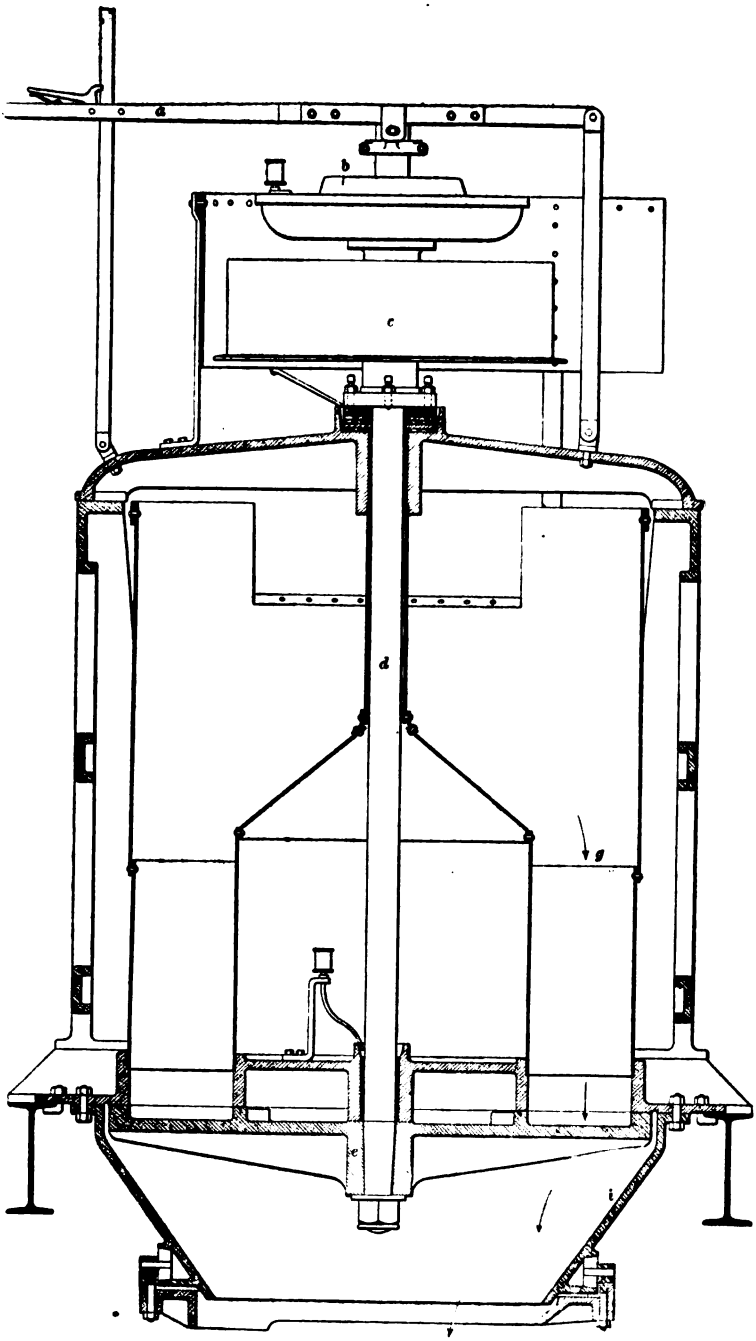

FIG. 9 (*b*)

which are accumulated in the space between the perforated false bottom *k* and the shell *a*.

19. Slicing the Beets.—At the present time, the diffusion process has replaced all others in the extraction of the juice from the beet. The first step in this process, after the beets have been thoroughly cleaned, is to cut the roots into thin slices, and it is of great importance that these slices be of uniform thickness. The machine in common use for this purpose is shown in Fig. 9 (*a*) and (*b*).

In the figure, *a* represents the lever for throwing the clutch *b* in and out of action; *c*, the driving pulley, that operates the shaft *d* carrying the circular frame *e*, which holds the knives *f*; *h* are pins used to keep the weight of beets off the knives, so that they may be replaced by new ones when dull without unloading the cutter. The beets fall into the cutter and, following the course of the arrows *g*, are cut by the knives *f* into **V**-shaped slices, which are usually 1/8 inch thick and 5/8 inch wide. These slices, or cossettes, fall through the hopper *i* into a chute, which conveys them to the diffusion-battery cells on the floor below. Some factories use knives that cut a rectangular-shaped cossette, but most modern factories prefer the **V** shape, as in the rectangular shape the walls are much thicker and a higher temperature is required to extract the sugar therefrom, with the ever-present danger of retarding the circulation of the juice through the cossettes by softening, or matting, them by excessive heat.

20. Many new factories are installing two cutters for each battery, thus being able to send an uninterrupted stream of cossettes to the battery, as a reserve cutter is always ready for use when knives become dull or broken, or in case a cutter for any reason gets out of order. Much trouble and delay have been caused by stones, iron, etc. being carried along with the beets and breaking the beet knives; but this difficulty is fast being overcome by the *stone catchers* of various designs that are being installed. The most successful are those placed in the bottom of the washers, or trash catchers, and are simply sheet-iron boxes kept filled with water, through which the beets flow on their way to the elevator, the stones falling to the bottom and being removed at regular intervals through a door in the side. Another is a large iron basket with perforated bottom placed under the beet flume; the top of the basket is on a level with the bottom of the flume and, as in the other case, the beets float on over this basket, the stones depositing in the same.

21. Diffusion.—In this process, the beets, after being shredded into small, thin strips, as described in the previous article, are brought into contact with water, and the sugar in the beet sap diffuses out through the cell walls while colloid substances remain in the cells. As the sugar is held in solution in the cells, and as these cells also contain most of the impurities, the cutting machine and knives must be selected with a view of cutting the beets in such a way as to rupture as few cells as possible and, at the same time, prepare a large surface for the action of the water or the dilute juice in the operation of diffusion without destroying the rapid circulation of the liquid. In the raw state, the protoplasmic lining of sugar cells will retain the sugar even if it be immersed in water, but, upon heating, a change will take place, and the cell that previously prevented the process of diffusion will readily permit the sugar to pass out into the water until the water on the outside of the cell contains the same percentage of sugar as that on the inside. If this water is then drawn off and replaced with fresh water, this process will again take place and may be continued until no more sugar remains. It might be thought that the diffusion process would yield a bulky diffusate, the evaporation of which, for the recovery of sugar, would be a costly operation, but this is not the case, as the diffusion is carried on systematically, and thus the quantity of water needed is reduced to a very reasonable limit.

22. The vessels used for diffusion are mostly upright iron cylinders, with flat or arched bottoms, having a large opening, capable of being tightly closed, for receiving the slices. A number of such diffusers connected together is called a *battery*. In order to keep the contents at the required temperature, diffusion not taking place at ordinary temperature, there is connected with each diffuser, or cell, a so-called heater, which reheats the juice before being admitted to the next diffuser in line. The vessels are connected in such a manner, by means of pipes, that the same portion of liquor can be driven through the entire battery. The driving power is hydrostatic pressure, and is obtained from a tank, or cistern, in the upper part of the sugar house, giving a pressure of from 15 to 25 pounds per square inch.

23. Circular Diffusion Battery.—Circular diffusion batteries are now in general use in this country, as well as in the European sugar-producing countries. A circular diffusion battery may contain from ten to fourteen diffusers; in Germany, as a rule, batteries from ten to twelve cells are preferred, while in this country the fourteen-cell battery is most universally used. A fourteen-cell diffusion battery, constructed by the Walburn-Swenson Company, of Chicago, is shown in Fig. 10.

It consists of fourteen diffusion cells C_1 to C_{14}, each of which is provided with a manhole on the top, which can be securely screwed down. Into each manhole cover is fitted an air cock Y, which serves as an air outlet and to ascertain whether the cell is filled. At the bottom of the diffuser is a drop door, which is closed by means of a hydraulic cylinder K (which receives its water through the water-line X), and a balance weight W. Each diffuser is connected with the corresponding heater by a short piece of piping, the one marked F, which connects diffuser C_{14} with the heater H_{14}, being the only one shown in the illustration. The heaters receive the steam through the steam line S, and the connection between the steam line and the heaters may be severed by means of the valves R. The heaters are connected with the liquor line B by means of a valve M, for drawing off or for

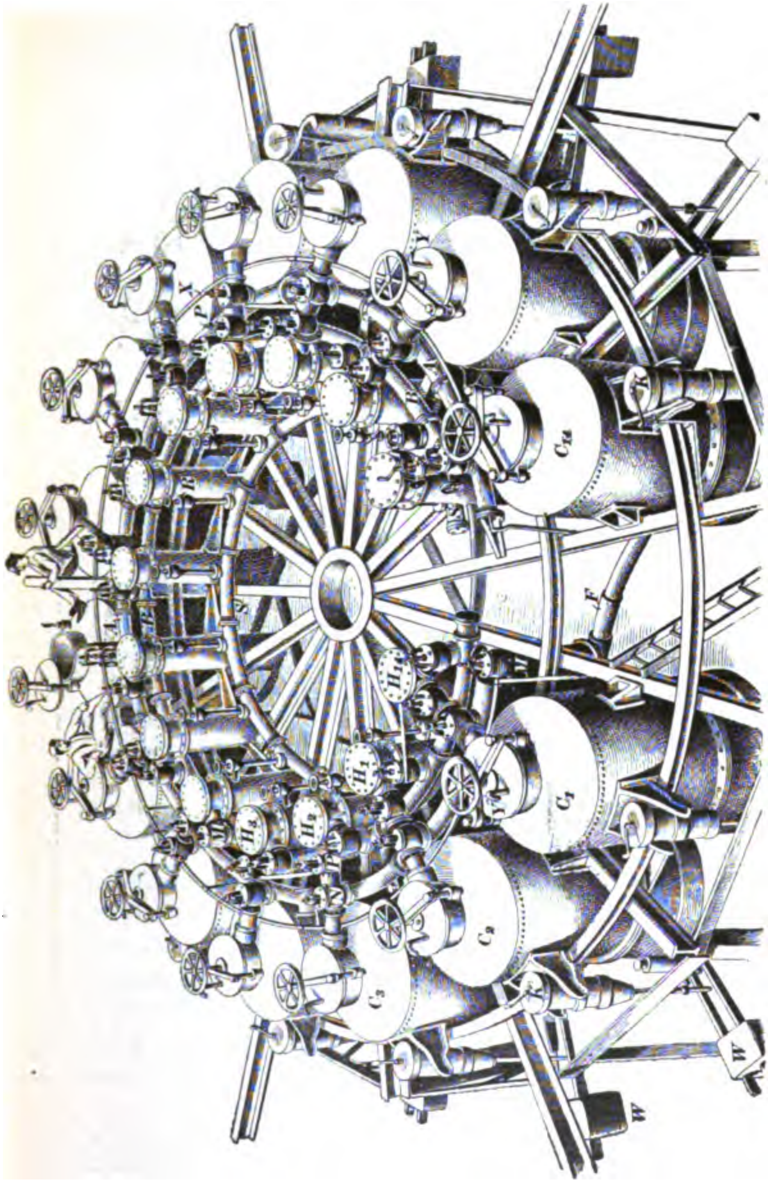

FIG. 10

circulation when some cells are cut off. N is the cross-valve for the water-line A, for taking the water into cell C_2 or into heater H_2 (and then into cell C_2) through the valve P. The heaters are provided with drain valves at the bottom, which are not shown in the cut.

24. In addition to the circular battery are also straight-line and double-line batteries. As the names indicate, the cells in one case are in a straight line; in the other, in two parallel straight lines. Precisely the same principle and operation are used as in the case of a circular battery. A late innovation is the doing away with the heater H (Fig. 10), the juice being heated on its way from one cell to the next by injectors placed in the pipes, thus materially simplifying the battery and avoiding the danger of juice leaking from the heater tubes into the drain lines.

25. Process of Diffusion. — The operation is carried on as follows: First from ten to twelve cells are filled with beet slices and water that has been previously heated to the desired point (say 80° C.) is admitted until the cell is filled. Assuming that the slicings contain 14 per cent. of sugar, there is an interchange of the sugar in the cell to the water outside until there remains 7 per cent. of sugar in the cell and 7 per cent. in the water; this water, now a dilute juice, is forced into the next cell. Having been heated in transit to 80° C., there is again an interchanging of sugar from the cell to the juice; this time the water admitted having already 7 per cent. of sugar will extract but half of the difference between the sugar in it and in the beet, and there will be 10.5 per cent. of sugar in both water and slices; this again is forced over to the next cell of fresh slices, and again there is an interchanging, or evening up, and there will be 12.25 per cent. of sugar in the juice, and so on until the water has passed over eight to ten cells, when it becomes nearly as concentrated as the beet juice.

To go back to the first cell: in this we had slices with 7 per cent. of sugar and 7 per cent. of sugar in the water; this water was forced over to the next cell by means of fresh water; this water again eliminated half of the sugar, and there remained but 3½ per cent. sugar; when the third was filled, it was done by forcing water into cell No. 1, which in turn being connected, forced the liquid content of cell No. 2 to cell No. 3, and so on. This fresh water that for the third time has been admitted to cell No. 1 again extracts half of the sugar, leaving 1.75 per cent. of sugar in both water and slices. This process is carried on until there remains from .5 to .25 per cent. of sugar in the slices, when they are emptied by means of the door at the bottom of the cell, and the cell is refilled with fresh slices to be again subjected to the same process.

26. Suitable Water for Diffusion Battery.—The water for the diffusion battery should be as pure as possible and should contain a minimum amount of calcium and magnesium salts, and of the salts mentioned below as *melassigenic*, i. e., molasses-forming. The calcium and magnesium salts, notably the bicarbonates and the sulphate of calcium, foul the heating surfaces of the battery. The bicarbonates decompose to some extent in the diffusers and deposit the normal carbonates upon the cossettes and probably influence the diffusion unfavorably. The water should not contain more than 10 parts per 100,000 of calcium sulphate, otherwise incrustations may form at some stage of the process. The most important melassigenic salts are sulphates, alkaline carbonates, and nitrates.

27. Temperature of Diffusion.—The higher the temperature maintained in the battery, other conditions being equal, the faster the diffusion. A low temperature requires a long contact of the slicings with the water, in order to obtain a satisfactory extraction. Many authorities limit the maximum temperature to 80° C. The eminent French authority *Dupont* recommends the following temperatures in a battery of twelve diffusers, ten of which are in activity:

Diffuser No.	1	2	3, 4, 5, 6, 7, 8	9	10
Temperature, deg. C.	40°	60°	76° to 85°	65°–70°	40°–50°

28. Difficulties in Diffusion Work.—Some difficulties may be experienced in the conduct of the diffusion when the supply of beets is irregular, the battery overheated, the roots imperfectly washed, or the knives improperly sharpened or set. The remedies are self-evident. The work should be so conducted that the juice may be drawn at regular intervals. The intervals should be lengthened in the event of a shortage in the supply of beets or other delays in the sugar house. A long delay in the diffusion work, or excessively slow work, results in impure juices, which yield but stubbornly to subsequent treatment.

Overheating affects the purity of the juice adversely and cooks the beet cuttings, causing them to pack or mat in the diffuser, and thus renders the circulation of the juice slow and imperfect and the pulp difficult to remove from the cell. Overheating may also cause pectic bodies to pass into solution, which later in the manufacture result in compounds that impede the filtration of the juice.

29. Measurement of the Juice.—The diffusion process requires that definite volumes of juice be drawn from the battery for definite quantities of beets. The juice is drawn into a measuring tank, which is alternately filled and emptied. If this measurement be made and reliable samples of the juice be drawn, a basis is supplied for subsequent control work. Unfortunately, this measurement as usually made is only approximate. Errors are introduced through variations in the temperature of the juice and the difficulty of closing the inlet valve of the measuring tank at the proper instant. Hence, special apparatus is essential to accurate measurement. This apparatus should be so arranged that it is wholly or partly automatic in its function. Whatever the system of tank measurements, it is essential that the measuring tank be carefully calibrated by means of a known volume of water rather than by calculation. A slight error in the calibration is multiplied many times before the end of the manufacturing season.

30. Automatic Recording Apparatus.—The errors mentioned above may be reduced to a minimum by a careful supervision of the battery temperatures and the use of automatic recording apparatus and overflow pipes.

The apparatus invented by Harsin-Déon, which is largely used in France, deserves mention. It consists essentially of a paper-covered cylinder revolved by clockwork. A float in the measuring tank is connected by means of a wire or chain with a drum, which revolves when the float rises or falls; or the shaft of the drum is a pinion, which, in revolving, engages a rack; this latter, in turn, is attached to a small arm, which carries a pencil. When the juice enters the measuring tank, the float lifts, revolves the drum, and, by means of the motion transmitted through the rack and pinion, the pencil traces a line on the paper-covered cylinder. The paper is divided vertically into 12 parts, corresponding to the hours. These parts are subdivided into 5-minute spaces. The cylinder makes one revolution every 12 hours. The sheet of paper is ruled horizontally into spaces of such width that each represents a certain volume of juice. It is evident from this description that the apparatus will record irregularities in the operation of the diffusion battery. As the lines traced by the pencil bear an invariable ratio to the depth of the tank, the volume of the juice may be deduced from the height of the *peak* of the curve above the base line. Bell signals, also operated by the float, warn the battery man when the tank is filled nearly to the required point, or is almost empty. A counter records the number of times the tank has been filled.

31. Calorisation.—From the measuring tank, the juice flows by gravity into a heater, technically known as a calorisator, where the juice is heated to between 85° and 90° C. This is done for the purpose of purifying the juice before carbonation, by coagulating the albuminous (nitrogenous) matter, which will not coagulate after the addition of lime in the carbonators, as albumin is soluble in alkalies. The above-mentioned temperatures should always be given the juice, for if it is not done, the impurities will be found in the molasses and will give trouble throughout the whole process. Although the really rational one, this practice is not generally adopted, and in many sugar houses lime is still added immediately after the juice leaves the measuring tank.

32. Carbonation.—As in the case of juice from sugar cane, it is necessary to purify beet juice before concentration, by treatment with lime, which neutralizes any acid substances that help to increase the inversion of the juice. In modern practice enough lime—about 3 per cent. of the weight of the beets from which the juice has been obtained—to convert the whole of the increase into calcium monosaccharate ($C_{12}H_{22}O_{11}CaO, H_2O$) is added, and the saccharate is decomposed as soon as it is formed by treatment with CO_2, which is blown into the vessel in which the defecation with lime is effected. It appears that the comparatively bulky precipitate of calcium carbonate that is thus formed carries down with it much of the coloring matter and various impurities, yielding a better juice than that obtained by employing a smaller quantity of lime, such as would suffice for neutralizing the organic acids naturally present in the juice. The process is carried out in a covered tank heated by closed steam (to avoid dilution of the juice) and is provided with a perforated delivery tube through which CO_2 can be blown in. The order of operation consists in the admission of the juice to be carbonated, the addition of lime, preferably in the form of milk of lime, and heating and carbonating the resulting mixture. Excess of CO_2 must be avoided, as carbonic acid is capable of causing a certain amount of inversion. The gas is therefore admitted until the alkalinity of the juice corresponds to 1 gram to 1.6 grams, calculated as lime CaO per liter of juice.

33. In the early part of the season, the quantity of lime used for the first carbonation is much smaller than the amount necessary later on. More lime is required when the beets are in bad condition than when sound. The quantity used is about 15 pounds of quicklime per 100 gallons of juice, but with unsound beets this amount is often exceeded.

In France, the lime is usually slaked and reduced to a milk of 20° Baumé with the thin juice obtained in washing the filter cake. The practice in many sugar houses is to place the quicklime in wire baskets and slake it directly in the juice in the carbonation tank. The carbonation should be effected with rich gas, that is, gas containing approximately 30 per cent. of carbon dioxide, thus not only economizing time, but producing a precipitate that is more easily removed by the filter presses. Practice differs as to the temperature of the carbonation, but, with rich juices, a temperature of about 85° C. appears to give satisfactory results. With weak juices, a lower temperature is often advantageously employed. Towards the end of the carbonation, the juice is heated from 80° to 90° C., thus breaking up the sucrocarbonates of calcium that have been formed. Practice also differs relative to this temperature.

34. Especial attention should be given the carbonation process, as it is the most important operation in the purification of the juice. When the carbonation is properly conducted, no trouble will be found with the juice or thick liquor further on in the operation; but in the event of poor or careless carbonation much trouble and annoyance will be encountered, as, for example, slimy cake in the presses, through which it is impossible to force the juice; heavy incrustation on the evaporator tubes, requiring numerous cleanings at very considerable expense; much sediment in the thick liquor, or pan supply tanks; trouble in boiling to grain; and a high percentage of sugar in the molasses. Proper temperatures during the carbonation process and a uniform, granular precipitate with proper alkalinity of the juice will greatly obviate all these troubles.

35. Second Carbonation, or Saturation.—To complete the defecation, the juice, after filtration from the sludge, is again treated with lime and the admission of CO_2 repeated; a liquid approximately neutral is thus obtained. The neutralization of the lime with CO_2 is essential, inasmuch as it would otherwise, on boiling, react with the albuminous substances contained in the crude juice and yield decomposition products instead of forming a precipitate and freeing the juice from impurities of the albuminous class.

The quantity of lime required in the second carbonation is from 2 to 4 pounds per 100 gallons of juice. The gas is passed into the limed juice, when working with sound beets, until a test shows that the free lime is saturated. Late in the season, or when the beets are in a bad condition, the supply of gas is cut off before the alkalinity is entirely saturated. When working without sulphur, the alkalinity is usually reduced to .02 gram per liter. On the completion of the carbonation, the juice is boiled 2 or 3 minutes and then filtered.

36. Difficulties in Carbonation.—A defective carbonation results in difficulty in the filtration of the juice, imperfect removal of the sugar from the filter-press cakes, juices of lower purity than necessary, and formation of lime cells. It may also result in difficulty in boiling the sugar to grain in the vacuum pan. These difficulties arise from a slow carbonation, too low a temperature, too little lime, excessive use of the carbon dioxide, or excessive alkalinity of the juice from the second carbonation.

37. The influence of the alkalinity on the second carbonation, or saturation, is great. The most experienced chemists in this line of work deem it inadvisable to allow the alkalinity to fall below .05, as otherwise some compounds, especially magnesia, will be released and pass again into solution, and cannot be eliminated in the third carbonation; these substances form incrustations on the evaporator tubes and cause loss of heating efficiency.

38. In Fig. 11 (*a*) and (*b*) is shown an illustration of a Dyer and Company carbonation tank, in which *a* represents the outlet valve for the diffusion juice; *b*, the outlet valve for the finished, or carbonated, juice; *c*, the valve for the

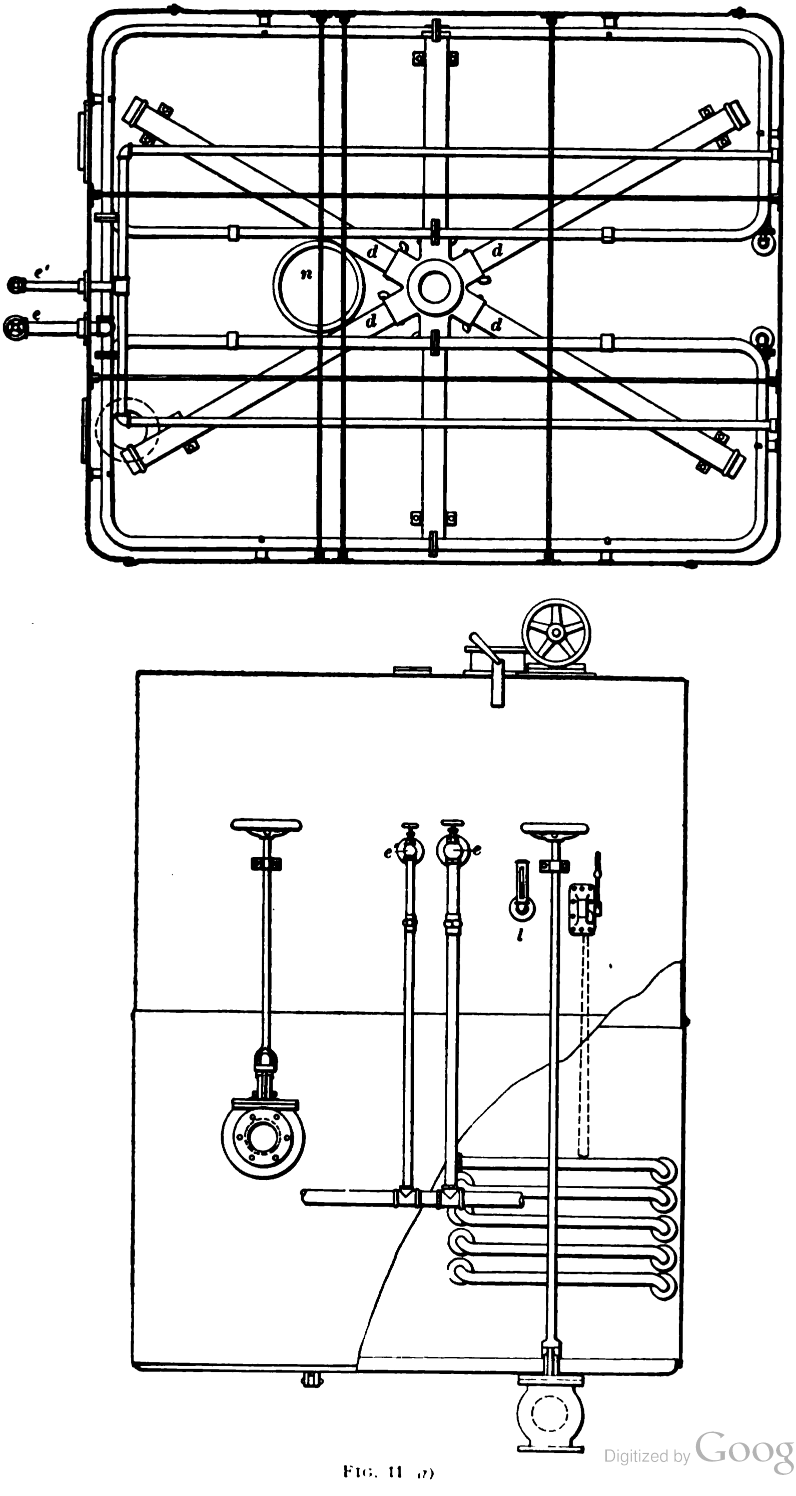
e'
e
n
d
d
d
d
e
e
l

FIG. 11 (a)

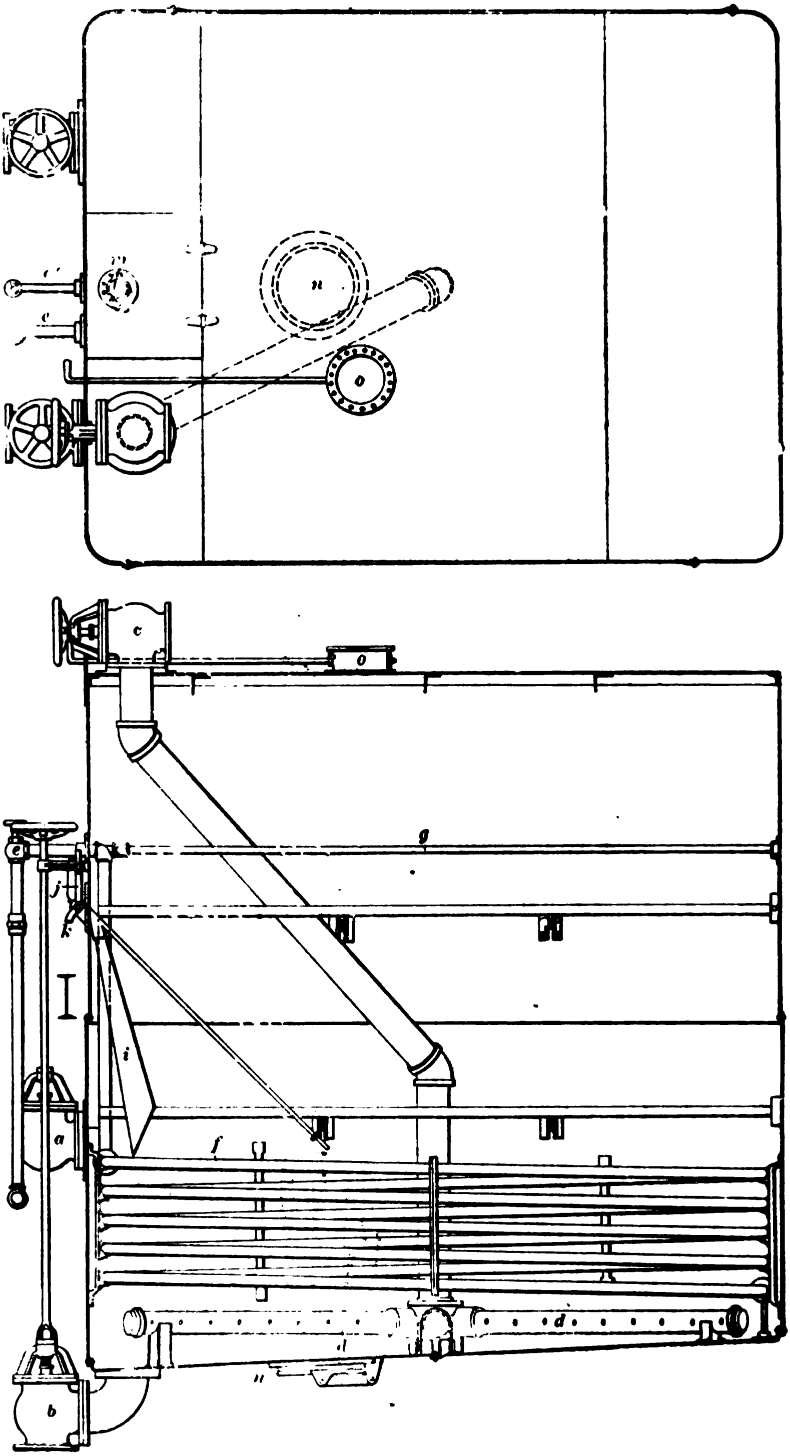
m
c'
n
c
o
c
o
g
e
j
k
I
i
a
f
d
d
n
b

FIG. 11 (b)

entrance of the CO_2, which enters the juice through the perforated pipes *d*; *e*, the steam valves supplying steam for the heating coils *f*, and for the perforated pipe *g*, the latter being used for blowing off the foam on the juice; *i* is the sampler for taking tests of the juice, which may be thus obtained without opening the tanks by simply pressing down on the handle *j* and holding a dipper under the spout *k*; *l* is a thermometer; *m*, a sliding cover; while *n* is a manhole in the bottom of the tank for cleaning out the same; *o* represents an opening to the vent pipe, which liberates superfluous gases to the outside air through a stack.

39. Lime Kiln.—The lime and carbon dioxide employed in the operation are usually made in the factory. The kilns are provided with collecting flues, through which the gas is drawn off. It is then scrubbed and freed from dust and SO_2. The latter is removed, not on account of its being objectionable as far as the sugar is concerned, but because it attacks the fittings of pumps that draw it from the kiln.

The relative quantities of coke and limestone used in the kilns vary between wide limits in the practice of various sugar houses, and depend also more or less on the construction of the kiln and the quality of the limestone. Theoretically, 6 pounds of coke is required to decompose 100 pounds of limestone containing 95 per cent. of calcium carbonate, which is approximately 1 volume of coke to 6 volumes of limestone. In practice, however, 1 volume of coke to 4 or 5 volumes of limestone are usually taken, and some authorities even advise 3 volumes of limestone to 1 volume of coke. The limestone and the coke should be well mixed, and distributed as evenly as possible in the kiln. The gas produced by the furnace should contain, as has been previously mentioned, approximately 30 per cent. of CO_2.

40. In selecting a suitable limestone, its physical condition as well as its chemical composition should be taken into account. The stone should be compact and hard, thus reducing the quantity of fragments and the risk of "scaffolding" in the kiln. Excessive moisture, that is, exceeding 2 or 3 per cent., in the stone reduces the temperature of the kiln when charging, involving an imperfect combustion and the production of carbon monoxide CO. Further, such stone breaks into small pieces under the influence of heat. It is claimed, however, that a small percentage of moisture, say 1 per cent., facilitates the decomposition of the stone. Magnesium is not objectionable in the kiln, but if the limestone contains silica, it forms fusible magnesium silicates at a high temperature and increases the danger of scaffolding in the kiln. It is further not desirable, as it introduces difficulties in the purification of the juice and forms an incrustation on the heating surfaces of the evaporation apparatus. The same objections apply, with equal strength, to calcium sulphate.

The objections to the presence of silicates are as indicated above, in the formation of fusible silicates of lime and magnesium. Part of the silica passes into the juice with the lime, retards the filtration in the presses, and coats the cloths of the mechanical filters, to their detriment. Silica also forms part of the scale on the heating surfaces. Less harm, however, results from this substance in hard limestone than from that in soft stone; hence, if the stone is hard and compact, a larger content of silica is admissible. When necessarily using stone of comparatively poor quality, the best coke obtainable should invariably be employed.

41. Difficulties in Lime Kilns. — The difficulties usually encountered in the management of lime kilns may be stated as follows: A limestone containing too much silicate will show a tendency to fuse, and, if overheated, will adhere firmly to the wall of the kiln. Stone in too small pieces, or stone and coke, or an excess of coke, will sometimes scaffold, or bridge. These conditions soon prevent the downward progress of the stone and lime. These difficulties are obviated by the use of a good limestone of suitable size, by properly mixing the stone with

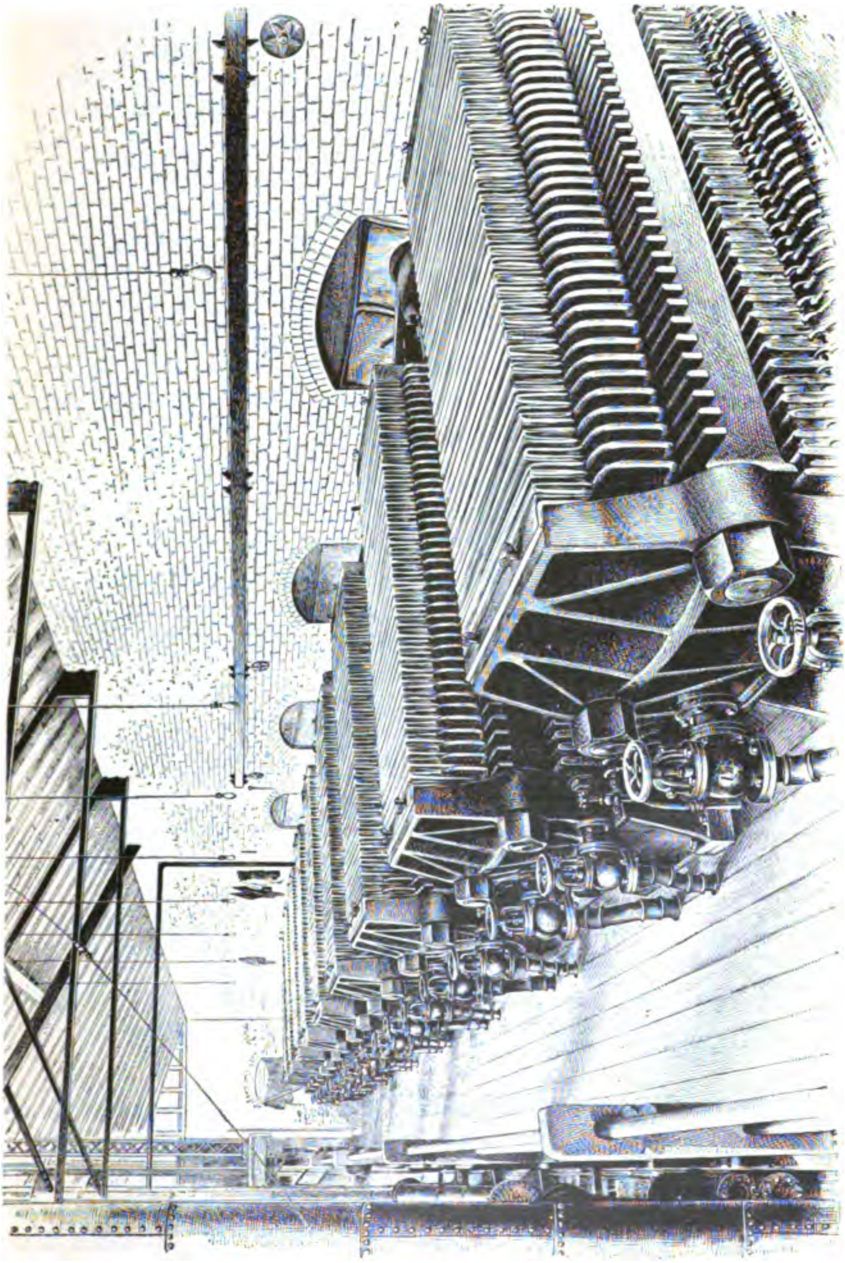

FIG. 12

the coke, by an even distribution of the mixture, and by a systematic withdrawal of lime. Should the charge scaffold in the kiln, it can only be broken down by the withdrawal of a considerable quantity of material at the lime doors and energetic use of an iron bar at the peep holes. The use of too little coke or too rapid withdrawal of lime results in an undue proportion of underburned, or raw, lime. The admission of too little air to the kiln results in an imperfect combustion and an excess of carbon monoxide in the gas. This carbon monoxide is not only a loss of carbon, but, if carelessly inhaled by the workmen, may result in serious poisoning. The addition of too much air dilutes the gas. This may result from leaking of the pipes, careless charging, or from running the gas pumps too fast.

42. Filter Pressing.—The removal of the sludge formed during the lime purification is effected in filter presses, a series of which is shown in Fig. 12, of such construction that the sludge can be washed and adhering sugar recovered. By this means, the content of sugar is reduced to 2 to 3 per cent. of the weight of the dried sludge, which amounts to about 10 per cent. of the beets treated. The sludge is composed of about 75 per cent. of calcium carbonate, and the balance consists of the calcium salts of the organic acids of the juice, small quantities of magnesia and alkalies, and a certain amount of insoluble organic matter. As it contains on an average of from 1 to 2 per cent. of P_2O_5, .3 to .5 per cent. of N, and .2 to .3 per cent. of K, it finds use as a fertilizer. If the manipulations of filter pressing have been carefully conducted, a treatment with SO_2 and filtering through sand are sufficient to insure a colorless, clear juice. In modern sugar houses, this plan is followed, but in some of the older and European factories a decolorization by animal charcoal is a rule.

43. Difficulties in Filter Pressing.—With juice from sound beets, properly treated in the carbonation process, the filtration is always easy and little difficulty is experienced. If the juice has not been sufficiently heated after

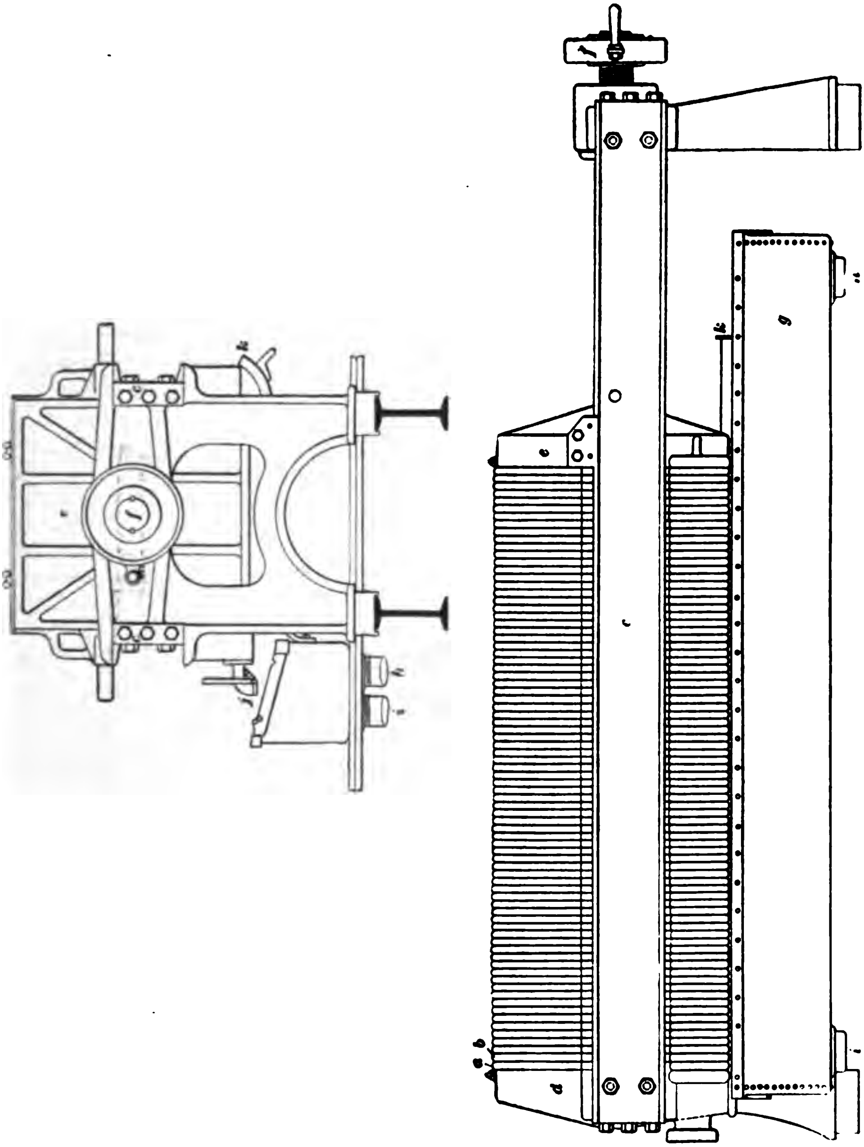
f
k
g
h
i
e
c
a
b
d
j

FIG. 13

the first carbonation, or the supply of gas has been cut off too soon, the juice will not filter well, just as if too little lime, or lime from stone containing too much silica or having hydraulic properties, had been employed. A proper adjustment of the quantity of lime added to the juice, and a careful control of the diffusion and of the first carbonation, will usually remedy difficulties in the filtration.

44. A detailed illustration of a modern filter press is shown in Fig. 13. It is composed of alternating solid and hollow frames, with small openings on either side, to allow the juice to pass from one to another, hung on two side beams, which, with the head- and tail-pieces, form a rack for supporting the frames. Referring to the figure, *d* is the head-piece; *e*, the tail-piece; *c*, one of the side beams; *f*, the screw; *g*, a trough for catching the filtered juice and wash water, and *h* and *i*, outlets, respectively, for the same; *j*, an outlet cock; *k*, an apron for catching any juice that may leak out between the frames.

In Fig. 12, *l* shows the outlet valve, for admitting juice to the press, and *m*, the water valve, for the admission of hot water, usually the condensed water from the evaporators, to wash the sludge or cake. In starting a press, the solid frames are covered with two thicknesses of a cloth manufactured especially for this purpose, and when these alternating solid and hollow frames are clamped together by means of the screw *f*, and the carbonator juice pumped into the press through the valve *l*, it is readily seen that the sludge or cake will accumulate on the cloth in filling the hollow frames and the clear juice run out through the cocks *j* into the trough *g*, thus being conducted on to the next operation.

When the press is full of cake, the juice valve *l* is closed, the water valve *m* is opened, and hot water is forced through the cake, thus washing out any remaining juice. The press is now opened by releasing the pressure of the screw *f*; the apron *k* is removed and the cake dropped through a hopper underneath and conveyed outside the building, to be used as fertilizer, or to be burned in rotary kilns for the recovery of the lime.

45. Sulphuring.—Sulphurous acid is usually employed in the gaseous state, in the manufacture of white sugar without bone black, and is usually applied by blowing the products of combustion from a small kiln burning sulphur directly into the juice. When sulphurous acid is used, the following procedure is advised : The second carbonation is stopped when the alkalinity is reduced to .5 to .6 gram per liter, calculated as lime CaO, and the juice is boiled and filter-pressed, as usual. Then the filtered juice is again treated with sulphur dioxide at a temperature of 95° C. until the alkalinity is reduced to .1 to .15 gram per liter; the juice is then boiled.

The sulphuring must be carefully controlled, in order to avoid loss through inversion, and the operation is performed in tanks of precisely the same construction as the carbonation tanks, the SO_2 being forced through the perforated pipes into the juice instead of CO_2, as in the carbonation process.

46. Concentration.—The juice purified so far is somewhat dilute and requires concentration. In this country it is concentrated in *multiple-effect evaporators*, the principle of which consists in the use of the steam from one pan, boiling under a pressure somewhat less than that of the atmosphere, to heat the liquid in the next pan boiling under still less pressure. A series of three vessels is known as a *triple effect*, and a series of four vessels a *quadruple effect*. In Europe, a triple effect has been found the most efficient arrangement, doubtful economy being obtained on extending the system to four or more vessels, although in this country the quadruple effect is generally preferred in the larger sugar houses.

The quadruple-effect evaporator is shown in Figs. 14 and 15 It consists of four bodies, each of which is arranged with steam chambers and tubes, with room for the vapors to disengage; the upper part, or vapor chamber, is connected

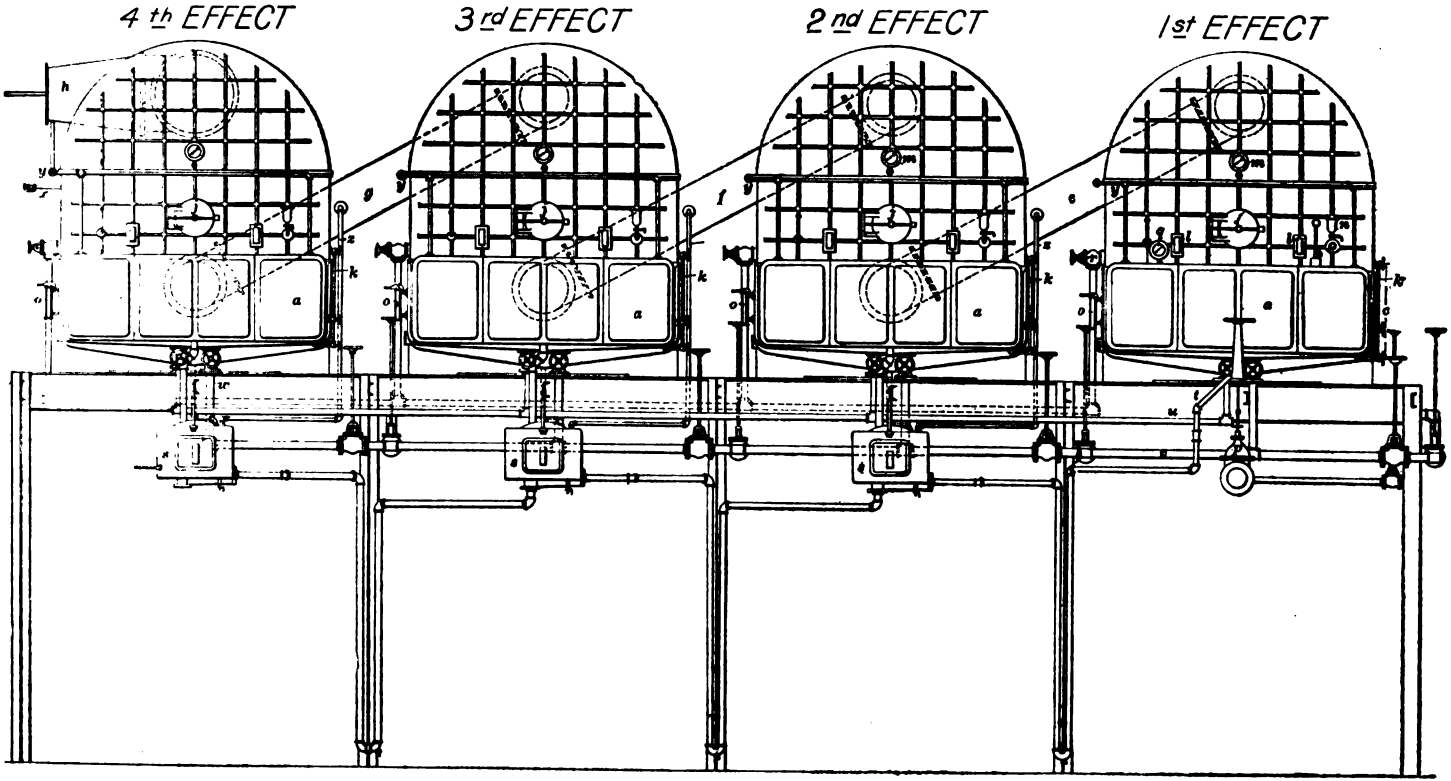
4 th EFFECT
3 rd EFFECT
2 nd EFFECT
1 st EFFECT
h
y
g
f
e
a
k
z
o
m
l
u
w

FIG. 11

with the steam chamber of the next body, so that the vapors from the boiling liquor may pass into the steam chamber of the next.

47. The operation of a quadruple-effect evaporator is as follows: The liquor space *c* (Fig. 14) of each body is filled with juice, and the exhaust steam from the different pump engines throughout the factory is collected in a pipe *d* (Fig. 15), which supplies the steam chamber *b* (Fig. 15) of the first effect with steam that causes the juice in it to boil; the boiling juice and the vapor from it have a temperature of about 100° C., which passes over through the pipe *c* (Fig. 14) to the next steam chamber and causes the

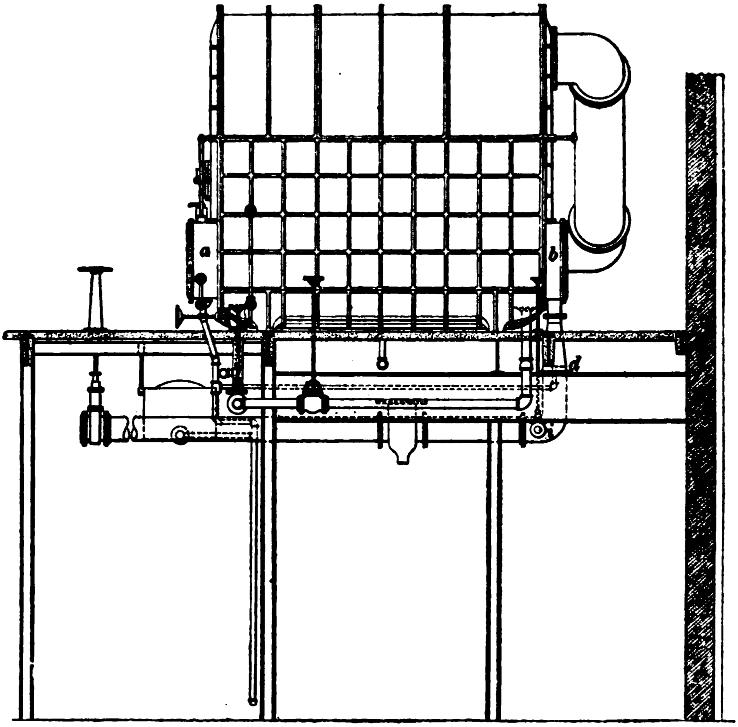

Fig. 15

liquor in this body to boil; the steam arising from it has a temperature of about 88° C. and passes over through pipe *f* (Fig. 14) to the next steam chamber, causing the juice in it to boil; the steam arising in the third effect and passing through pipe *g* (Fig. 14) over to the fourth effect has a temperature of 76° C. and boils the liquor in this body. The difference in temperature at which the liquor boils in each body is caused by the difference in pressure, which is maintained by means of a condenser and vacuum pump; the vapor arising from the last effect, or body, passes to a condenser through *h* (Fig. 14), producing a vacuum in the fourth effect of about 26 inches, the liquid in the last effect thereby boiling at 60° C. The fourth effect acts as a condenser and produces a vacuum to the third, the third to the second, and the second to the first. The first effect is connected to the live-steam line *i* (Fig. 15), so that in case the exhaust steam is not sufficient to evaporate the juice rapidly enough, live steam may be turned on to the first effect. Many sets of evaporators have a live-steam connection to each of the four effects, to be used when starting up with cold juice in all the bodies.

In Fig. 14, *j* shows a manhole door; *k*, glass columns, to show the height of the juice in each effect; *l* represents sight glasses, to watch the juice while boiling; *m*, vacuum gauges; *n*, so-called *butter cups*, which serve to introduce small quantities of oil, in order to keep down the foam of the boiling juice; *o*, sample cups for obtaining tests of the liquor. These testing cups are especially used on the fourth effect, to ascertain whether or not the liquor is of sufficient density (usually 30° Baumé) to be drawn off, which is accomplished by means of a tail pump on the floor below through the pipe *w*. The other accessories of a quadruple-effect evaporator, in Fig. 14, are: *p*, exhaust steam valve; *q*, pressure gauge on front of the steam chamber of the first effect; *r*, valve on water-line for cutting off effects when cleaning them; *s*, traps for condensed water; *t*, pipes leading water from one tap to another; *u*, the sewer line for draining off wash water; *v*, the juice line from one effect to another; *x*, an equalizer line connected with the thick-liquor tail pump, to equalize the vacuum in the pump, and in the fourth effect, in order that the liquor may be drawn from the effect while still under a high vacuum; *y* are equalizer lines for the steam chamber, and *z* are equalizer lines for steam traps.

48. Vacuum Pan.—The juice when sufficiently concentrated is run into the vacuum strike pan. This pan is a closed cast-iron vessel about 11 feet in diameter and 14 feet high, holding from 30 to 35 tons of *melada* (sugar and molasses) when full. In the lower portion are situated a number of copper coils, through which the steam passes without coming into contact with the boiling mass; the rising vapor passes to the condenser and a pump draws off the non-condensable vapors. During the operation, a vacuum is maintained in the apparatus, the mass boiling at a low temperature, which prevents burning.

The operation is as follows: Juice is admitted to the pan, and when about half full steam is turned on and the contents evaporated until it is "just about sugar." At this moment, fresh juice is admitted and the whole mass thinned, but not enough to dissolve the microscopic crystals; it is again allowed to concentrate by evaporation, thickened, and the sugar in the freshly admitted juice will join the microscopic crystals, which, increasing in size, will now be apparent to the eye. The mass is not allowed to concentrate enough to form more microscopic crystals, but again thinned by adding fresh juice before that point arrives which causes the crystals to grow in size without forming new ones; this operation of concentrating and diluting the mass goes on until the strike pan is full, the size of the crystals and the hardness depending on the way in which the operation is conducted.

49. Granulation of Sugar in the Vacuum Pan.—An excessive alkalinity or an excess of lime salts in the sirups causes the strike to boil slowly or heavily. When the difficulty is due to the alkalinity, the addition of sufficient dilute

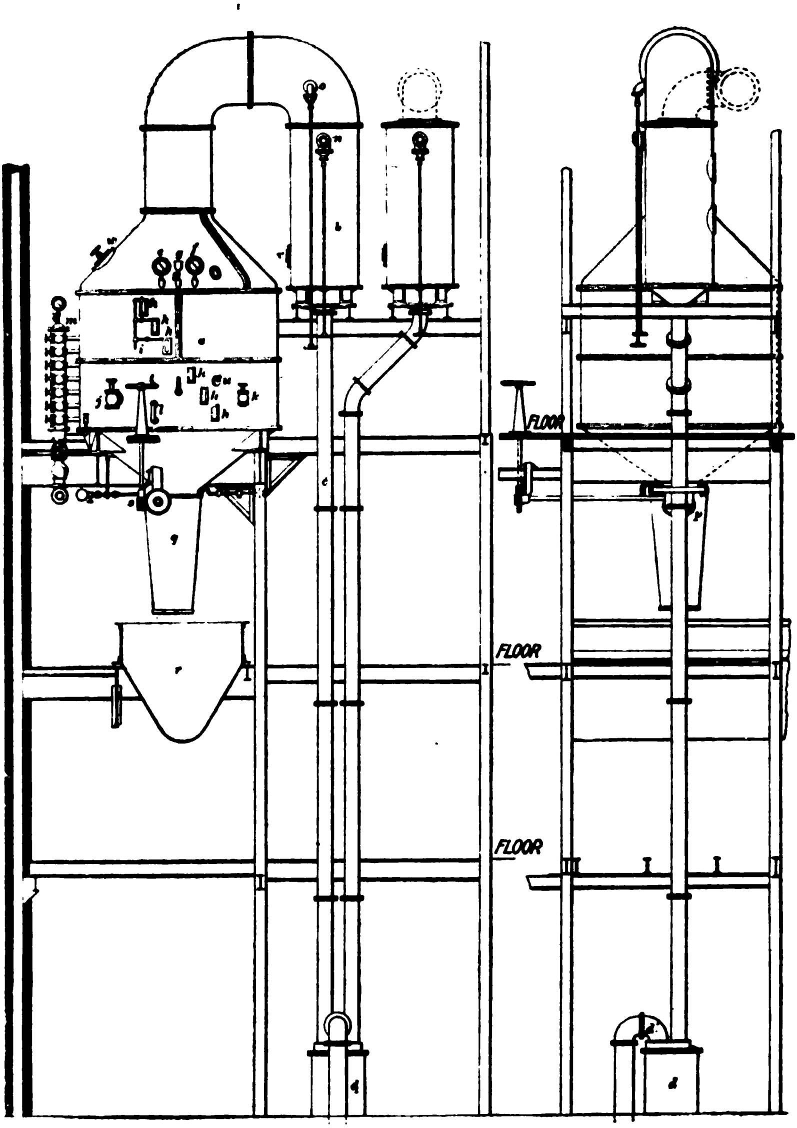
FLOOR
FLOOR
FLOOR

FIG. 16

hydrochloric acid to nearly neutralize the *masse cuite*, and careful supervision of the saturation with carbon dioxide or sulphur dioxide, is usually a satisfactory remedy. Excessive alkalinity of the sirup may be corrected by the addition of superphosphate of calcium, followed by filtration, to remove the precipitate, or the sirup may be nearly neutralized with dilute hydrochloric acid. The former method is preferable.

Should the difficulty be due to excess of lime salts, they may be decomposed by the addition of a vegetable oil or carbonate of sodium. The purification of the juice should be carefully controlled, so that it may seldom be necessary to adopt one of the remedies.

50. E. H. Dyer and Company's vacuum pan of latest design is shown in Fig. 16, *a* being the body of the pan; *b*, the condenser for producing the vacuum by condensing the vapors of the boiling liquor; *c*, a fall pipe for conveying the condensed and used water to the seal tank *d*, from where it runs by way of the pipe *d'*, with water from the evaporator condenser, to the beet sheds, to float the beets into the factory. The vacuum pan is further furnished with thermometer *e*, a vacuum gage *f*, a butter cup *g*, and sight glasses *h*. The inlet valve *j* is used for feeding thick liquor to the pan; *k* represents the water-inlet valve; *l*, the testing, or sample, cup; *m*, manifold of steam valves, to introduce live steam for boiling; *n*, water-inlet valve for the condenser; *o* is the vacuum breaker, allowing air to enter the pan and thus destroying the vacuum when the boiling is finished; *p* is a foot-valve for dropping the finished mass through the spout *q* into the mixer *r*, from which it is drawn in small amounts into the centrifugal machines on the floor below; *s* and *t* are gear and valve stems used for opening and closing the foot-valve *p*; *u* is a proof stick for taking and examining samples; and *v* and *w*, manhole covers.

51. Centrifugals.—The machines consist of revolving baskets surrounded by safety curbs. The baskets are lined with a finely perforated brass screen *a*, Fig. 17. About

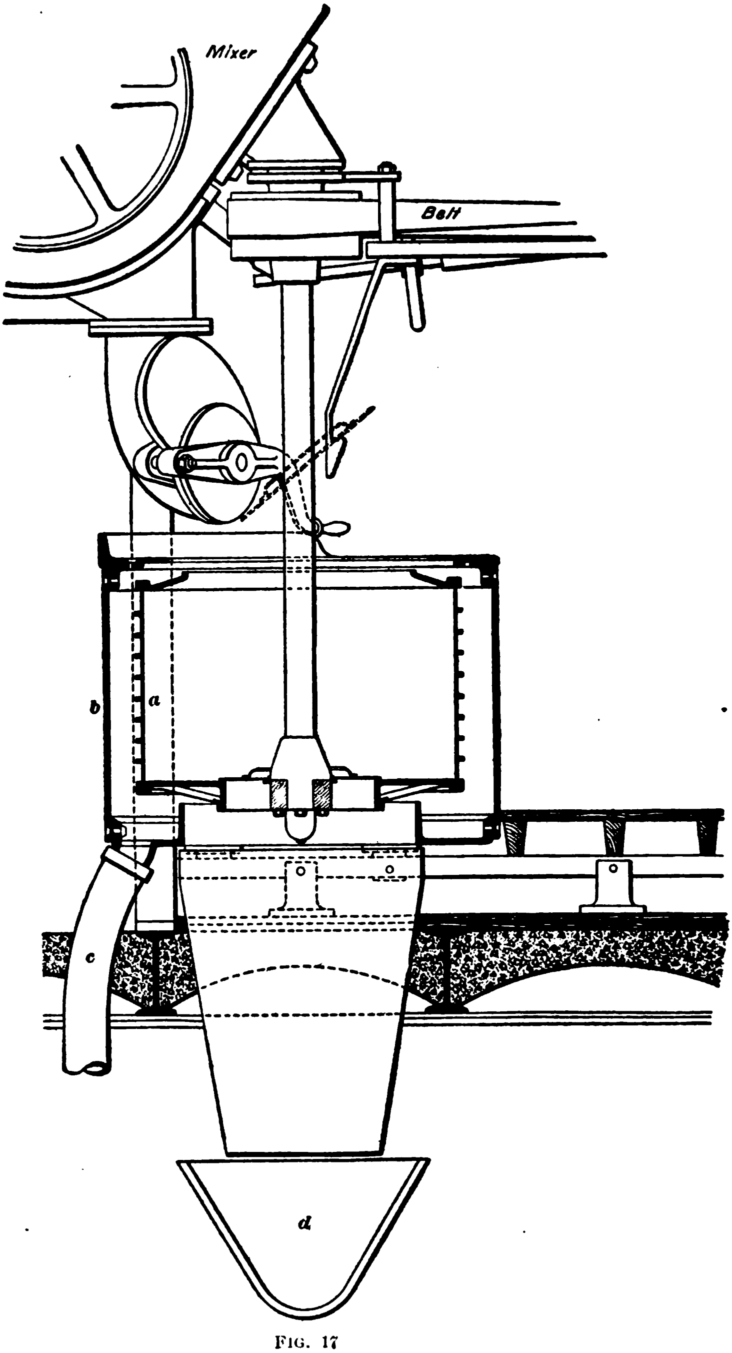
Mixer
Belt
b
a
c
d

FIG. 17

500 pounds of the magma is taken into a centrifugal from the mixer above at a time; the machine is caused to revolve by means of suitable belts and pulleys until it attains a velocity of from 1,000 to 1,500 revolutions per minute; the centrifugal force, which is about 40 pounds per square inch, throws the magma to the sides, the screen *a* holding the sugar back, and the molasses is thrown off through the screen until it strikes the sides of the safety curb *b*, flows off in a pipe *c* at the bottom, and is collected in tanks for further manipulation. The sugar crystals, worked free from molasses, fall through an opening in the bottom of the centrifugal (when the machine is stopped) into a conveyer *d* and are carried to a drier, consisting of a hollow cylinder about 6 feet in diameter and 30 feet long, which revolves slowly. The sugar being carried upwards and dropped continually on warm pipes is thus thoroughly dried and then screened, to separate any lumps, and dropped through a hopper, to be sacked or barreled ready for the market. In sugar houses having sufficient water-power, the water is used to run the centrifugals, as it is found to be the cheapest power, when abundant.

52. The molasses from the magma is again boiled in the vacuum strike pan and another crop of sugar, so-called *second sugar*, obtained from it, and also another lot of molasses. This second lot of molasses is, as a rule, too poor in sugar to be again crystallized in the vacuum pan; it is, however, concentrated to what is called *string proof* in the vacuum pan, and dropped, when finished, into crystallizers, where it is continually agitated, which, with careful attention to temperature, causes all sugar that can be obtained to crystallize. This sugar is known as *third sugar*.

53. Crystallizers.—The standard crystallizer mentioned above is represented in Fig. 18. The strike is dropped into the crystallizer *a* through the opening *b* and kept slowly in motion by means of the screw *c* until the sugar has crystallized sufficiently, when it is drawn off through the manhole *d*

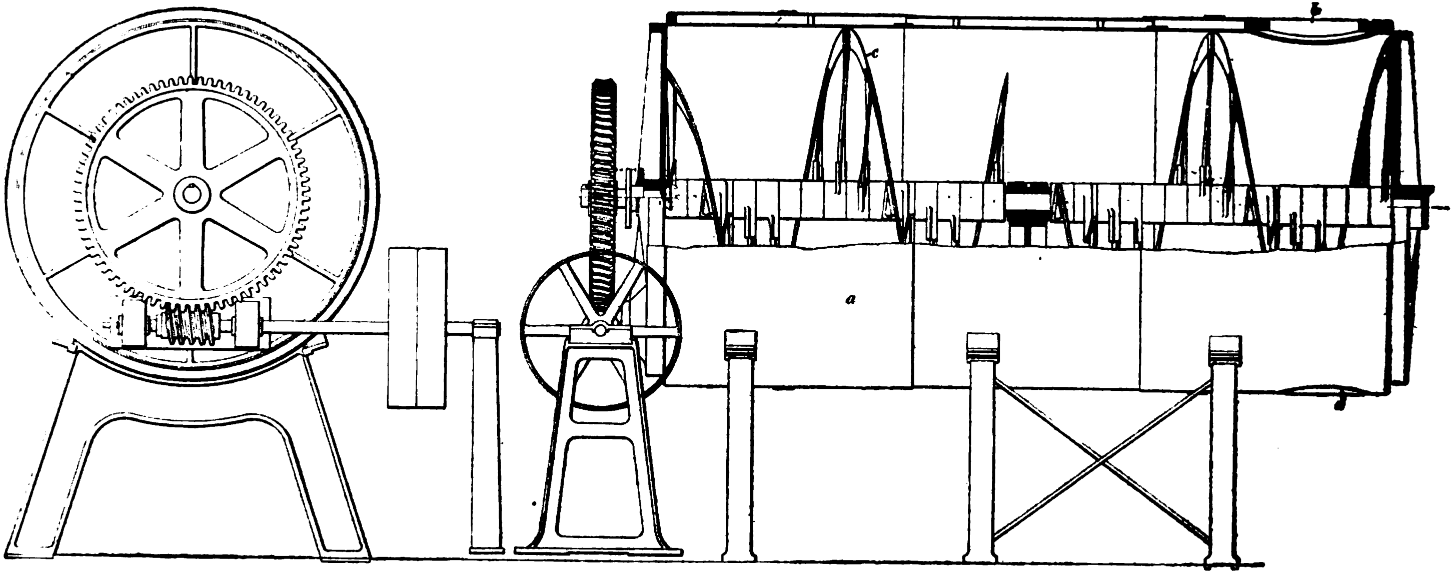

FIG. 18

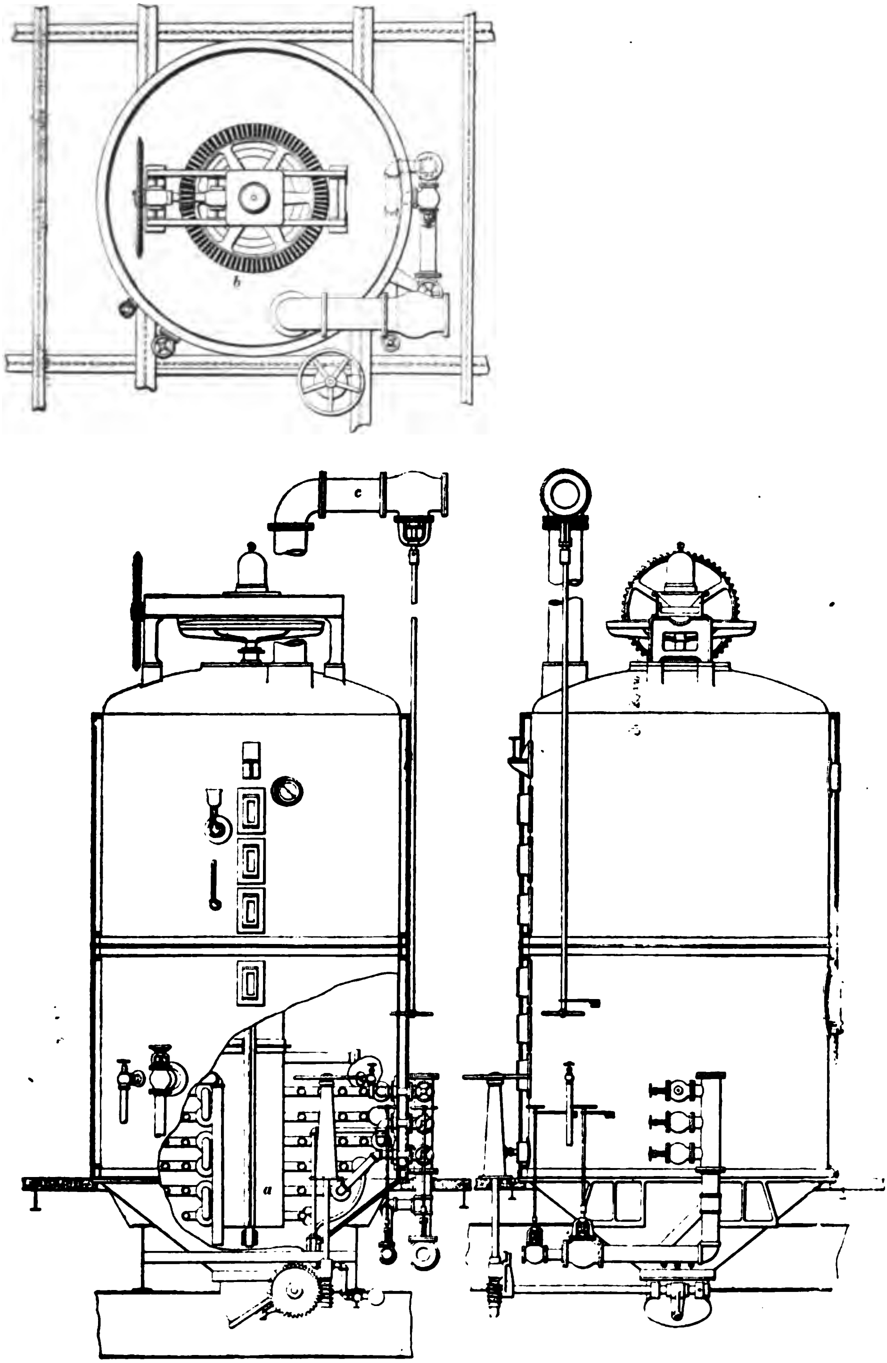

Fig. 19

and dropped into a second, or brown, sugar mixer and then worked in a second set of centrifugals. The second and third sugar, freed from their impurities, which remain in the molasses, are melted with thin juice and, being mixed with thick liquor, sent to the vacuum pan and reconverted into first sugar.

The Grossé vacuum crystallizer, shown in Fig. 19, is used in a number of new factories in place of the one shown in Fig. 18. In construction, it resembles the vacuum pan, but differs from it in having a screw within the pipe *a*, which carries the heavy mass from the lower part of the crystallizer and brings it continually to the upper part of the crystallizer, where it falls upon an umbrella-shaped plate and drops back again into the body of the mass. The object of this movement is to bring every part of the mass continually under the influence of the vacuum, thus expediting greatly the crystallization and producing a greater percentage of crystals than is obtained in the apparatus shown in Fig. 18, and in considerably shorter time. The similarity of this crystallizer to the previously described vacuum pan obviates the necessity of a description further than stating that *b* is the gear used for operating the helix, or screw, with the crystallizer and *c* the vapor pipe leading to a condenser, which is identical with that of the pan condenser in construction.

54. There is occasionally a tendency in molasses boiled to string proof to foam in the crystallizing tank, or, as this is usually termed by the workmen, to *boil over*. This has been attributed to various causes. It is often charged to reducing the alkalinity of the juice in the first carbonation too low. Caustic soda may be used to increase the alkalinity. Overheating of the molasses in the pan, or in the hot room, is supposed to often be the cause of the difficulty. The usual remedies are to sprinkle water or caustic-soda solution on the surface of the molasses.

55. The following analysis represents the average composition of beet molasses:

Sugar	50%
Other organic matter	20%
Ash (mineral matter)	10%
Water	20%
Total	100%

All the sugar that can be profitably removed from beet molasses by the ordinary process of concentration and crystallization has already been taken out, the mineral and organic matters preventing the separation of that still remaining. As already stated, cane-sugar molasses is edible, while beet molasses is very dark in color, and has an exceedingly disagreeable taste and odor. Choice between utilization for sugar by some other process, or spirit making, depends largely on the fiscal regulation of the country in which the manufactory is situated. Many attempts of more or less successful character have been made to recover the sugar from beet molasses, and a short account of the most important of these will now be given.

56. Osmose Process.—This process depends on the fact that when molasses is separated from water by an osmotic membrane—e. g., parchment paper—the salt in the molasses diffuses more quickly than the sugar into the water. The fact that a molecule of salt is smaller than a molecule of sugar will explain why salt will osmose more readily than sugar, for it is a fundamental principle in chemistry that elements dialyze (or osmose) according to their molecules, the smaller dialyzing more readily than the larger. A portion of the sugar left in solution can be recovered by evaporation. Some dilution from the incoming water takes place under these conditions, but the process has the advantage of simplicity and of requiring no addition other than water to the molasses to be treated. The organic matter (other than sugar) present in the molasses diffuses even more slowly than the sugar and remains in the liquor sent for concentration. Its presence hinders the separation of the whole of the sugar and necessitates the return of the molasses produced by concentration and subsequent centrifugal separation to the osmose apparatus. Three osmotic treatments of the same portion of molasses are as many as can be profitably performed, about half the total sugar in the molasses being thus recovered.

The process is carried out in an apparatus somewhat resembling a filter press in structure, consisting of a series of compartments separated by septa of parchment paper. The molasses is fed into alternate compartments and the water into the intermediate divisions. The weak saline solution obtained by the passage of the water through the second series of compartments is usually too dilute for use save as liquid fertilizer.

57. The object of the osmosis process (dialysis), as has been stated, is the reduction of the proportion of the saline and organic impurities, so that an additional quantity of sugar can be removed from the molasses by crystallization. The proportion of saline matter in the molasses and in the by-products from the osmosis is so high that the apparent coefficients of purity are of but comparatively little value and the time required for the determination of the true coefficients is so long that they cannot be made available for the immediate control. Notwithstanding the objections mentioned, manufacturers are compelled to be largely guided by the apparent purities in conducting the osmosis. The saline coefficient is a most reliable guide, but, unfortunately, its determination also requires much time.

In actual practice, the following analytical determinations are usually made in the molasses, before and after osmosis, and in the osmosis water: Degree Brix, percentage of total solids by drying, percentage of sucrose, percentage of ash, percentage of organic matter not sucrose by difference, percentage of reducing sugars, and alkalinity due to lime. The following coefficients, true and apparent, should be calculated: Coefficient of purity, saline coefficient, glucose coefficient, and coefficient of organic matter. Practice, and the expense of the process, as compared with the value of the sugar recovered, must be the guides in determining the improvement to be made in the above coefficients.

Gallois and Dupont in their *Manual-Agenda* give the following advice relative to the character of molasses that may be treated by this process with profit: "It is useless to dialyze molasses whose saline coefficient is higher than 6, since it will yield a satisfactory quantity of sugar on further concentration. Molasses containing more than 1 per cent. of reducing sugar cannot be treated with profit. Molasses containing much lime, especially organic salts of calcium, are difficult to dialyze. Such molasses should receive a preliminary addition of sodium carbonate or acid barium phosphate to precipitate the lime, which must be removed. Molasses containing as much as .2 per cent. of lime CaO should be treated as indicated above. If there are indications of fermentation, or if the molasses is but slightly alkaline, neutral, or acid, caustic soda should be added."

58. Triple Osmose Process.—The triple osmose process, invented and controlled by Henry A. Vallez, is being installed in many of the American sugar houses and seems certain to continue as a great success in this country when we take in consideration the facts that our molasses is worthless and that the quantity of molasses produced here is fully 50 per cent. greater than in European countries, on account of the large quantities of salts contained in the beets.

A molasses with a composition of 50 per cent. of sugar and 15 per cent. of ash will, by osmosing, lose about 10 parts of sugar and 10 parts of salts; thus the molasses, after osmosing, will contain 40 per cent. of sugar and 5 per cent. of ash. As 1 part of ash prevents 3½ parts of sugar from crystallizing, it will be seen that the molasses before treatment would be practically worthless for sugar, but after the treatment, we find that the 5 per cent. of ash will only prevent 17.5 per cent. of sugar from crystallizing, leaving 22.5 per cent. of sugar free to crystallize.

59. Definitions of the Coefficients.—The terms *coefficient of purity, true and apparent, saline coefficient*, and *glucose coefficient* have been mentioned in the previous article, and will now be defined.

The **true coefficient of purity** is the percentage of sucrose contained in the total solid matter in the product, and is calculated by dividing the percentage of sucrose by the percentage of total solids, as determined by drying, and multiplying the quotient by 100.

The **apparent coefficient of purity** is calculated as above, except that the degree of Brix, as determined by spindling, or from the specific gravity, is substituted for the percentage of solids, as ascertained by drying.

The coefficient is also often termed the *quotient of purity*, the *degree of purity*, or the *exponent.*

Glucose coefficient, or **glucose per 100 sucrose,** is also frequently termed the *glucose ratio.* Thus,

$$\frac{\text{Per cent. reducing sugar}}{\text{Per cent. sucrose}} \times 100$$
$$= \text{glucose (reducing sugars) coefficient.}$$

This coefficient is useful in detecting inversion. An increase in the glucose coefficient at different stages of the manufacture, provided there has been no removal of sucrose or decomposition of reducing sugars, shows that a portion of the sucrose has been inverted.

The **saline coefficient** is the quantity sucrose per unit of ash. Thus,

$$\frac{\text{Per cent. sucrose}}{\text{Per cent. ash}} = \text{saline coefficient.}$$

60. The property possessed by sucrose of forming compounds—saccharates or sucrates—with calcium oxide, strontia, and baryta is turned to account in various processes for the recovery of crystallizable sugar from beet molasses. The following are typical methods of this class, but are not, as a rule, employed in the United States.

61. Elution Process.—The molasses is mixed by means of an edge runner with about 25 per cent. of its weight of quicklime, but little reaction taking place as long as the mixture is kept cool; on this account, the pan of the edge runner is artificially cooled. When the mixture is complete, the mass is transferred to small iron chambers, in which its temperature rises spontaneously to about 100° C., and a combination of the sugar and the lime to form tricalcium saccharate $3CaO, C_{12}H_{22}O_{11}$ takes place, accompanied by expulsion of the surplus water and the formation of a hard, porous mass. This product is broken up and extracted systematically with weak alcohol (35 per cent.), impurities being dissolved and the calcium saccharate left fairly free from foreign matter. The extraction is carried out in an apparatus resembling a battery of diffusion cells. The alcoholic extract is distilled and the alcohol recovered, the aqueous residue being used as a fertilizer. The alcohol left clinging to the calcium saccharate is removed by steaming and is recovered. During the steaming, the tricalcium saccharate is partly decomposed, yielding monocalcium saccharate $C_{12}H_{22}O_{11}, CaO$ and lime. The complete separation of the lime can, of course, be effected by treatment with carbon dioxide, but the monocalcium saccharate is often substituted for lime, in the early stage of the winning of raw sugar, for neutralizing the acidity of the diffusion juice; the acid thus neutralized of course liberates an equivalent quantity of sugar, which is worked up with the bulk. Various modifications of the elution process have been devised, differing chiefly in the manner in which calcium saccharate is produced, but their underlying principle is identical with that enunciated above.

62. The **Steffens process** is distinguished from its congeners by the fact that the use of alcohol is dispensed with. It depends on the solubility of tricalcium sucrate at low temperatures. The molasses is diluted with water until its content of sugar is between 7 and 8 per cent., and quicklime is added to it in small portions, the total quantity being about equal to the weight of the sugar present. The liquor is well stirred during admixture, to prevent the temperature from rising above 15° C. At first, the lime goes into solution, but eventually it separates again as tricalcium saccharate accompanied by excess of lime. The mass thus obtained is filter-pressed, and the saccharate is used instead of lime in purifying the crude juice, and the washings are used for diluting the next batch of molasses.

63. Strontia Processes.—The process that at the present time is attracting very favorable attention in Europe, and seems to give considerable promise, is the **strontia process.** Several such processes have been devised, notably by Scheibler, in which strontia is substituted for lime in recovering sugar from beet molasses. The earlier of the processes due to this chemist consists in treating the molasses with strontium hydrate in sufficient proportion to form distrontium saccharate $2(SrO), C_{12}H_{22}O_{11}$, and to leave an excess of strontium hydrate, in a solution of which the saccharate is practically insoluble. The precipitate is thrown on a filter cloth supported by a perforated plate and stretched over a horizontal semicylindrical vessel. The air beneath the filtering cloth is exhausted and the desugared liquid pulled through. The distrontium saccharate is washed with a hot solution of strontium hydrate, turned out into iron boxes, by inverting the filter, and heated with water at a temperature below 15° C.; cold-air chambers are requisite in warm weather for this part of the process. The saccharate is decomposed, giving crystals of strontium hydrate and a solution of sugar, which is freed from the residual strontia by treatment with CO_2 and concentrated in a vacuum pan in the usual way. That part of the strontium hydrate that crystallizes is immediately available for further use, while that precipitated as carbonate is causticized by burning in a special kiln.

A more recent modification by Scheibler depends on the formation of monostrontium saccharate $SrO, C_{12}H_{22}O_{11}$, when a solution containing about 25 per cent. of sugar is treated with strontium hydrate at about 70° C. A slight excess of strontium hydrate is used and the temperature is maintained above that at which the formation of distrontium saccharate takes place. The instability of monostrontium saccharate is such that the addition of a crystal of strontium hydrate determines the precipitation of the same hydrate, and that of a crystal of monostrontium saccharate causes the salt to be thrown down. To the liquid, therefore, is first added a crystal of monostrontium saccharate and the precipitate filtered off; the filtrate is then boiled with strontium hydrate, yielding distrontium saccharate as a precipitate, which is used instead of strontium hydrate for treating a further portion of the molasses, another crop of monostrontium saccharate being thus obtained. The total yield of monostrontium saccharate is decomposed by dissolution in water, thorough cooling, and the addition of a crystal of strontium hydrate. The recovered strontium hydrate can be used again in the process and the sugar is freed from remaining strontia by means of carbon dioxide. The filtrate from the distrontium saccharate obtained above yields on crystallization crude strontium hydrate; residual strontia is recovered as carbonate, and the final filtrate contains the impurities of the molasses.

64. Preparation of Strontia.—The strontium hydrate used in this process is generally prepared in the sugar factory by burning native strontium carbonate (strontianite) to strontia, much in the same way as limestone is burned. A gas furnace is preferable for this purpose, as the comparatively expensive strontia is thus not contaminated with the silicious ash of solid fuel, and for a like reason the furnace should be basic lined. Strontia, being a more powerful base than is lime, has a stronger tendency to act on acid material (i. e., silicious bricks) and also requires a higher temperature for the decomposition of its carbonate. The strontium carbonate obtained as a byproduct in the process for winning crystallizable sugar from molasses is taken from the filter press, molded into bricks, and burned in the same manner as the native carbonate. The strontia is hydrated and dissolved in water, the hot solution, containing about 13 per cent. of SrO, being run off and allowed to crystallize, nearly the whole of the strontia being deposited in the hydrated state as $Sr(OH)_2 8H_2O$.

PRODUCTS OF MANUFACTURE

65. Raw Sugars.—The composition of the juice from both the sugar cane and the sugar beet has been stated and the processes for preparing the raw sugar from each of these sources. We may now examine more closely the character of the products obtained. The raw cane sugar, made as it is chiefly in the tropics under various conditions of working from the most primitive to the most improved, has come into commerce under a great variety of names, as well as varying grades of purity. The beet sugar as made in this country is usually very pure and is usually known as *first*, *second*, and *third sugar.*

Muscovado is brown sugar produced in the West Indies, generally by open-pan boiling, and which has been drained in hogsheads or perforated casks, and so freed in large part from the accompanying molasses.

Cassonade is a name formerly applied in the French colonies to muscovado sugar.

Melada is a moister brown sugar, produced like the muscovado, but not drained free from molasses.

Concrete, or **concreted sugar,** is a compact, boiled-down sugar, containing both the crystallizable sugar and impurities that ordinarily go into the molasses.

Clayed sugars have been freed from the dark molasses by covering them in molds by moist clay, which allows of a gradual washing and displacement of the adhering sirup.

Bastards is the name given to an impure sugar obtained by concentrating molasses and allowing it to crystallize slowly in molds.

Joggery is the name given to a very impure East India palm sugar, sometimes refined in England, but chiefly consumed in the country of its production.

Demarara crystals are the products of the best vacuum-pan boiling and have been well purged in the centrifugals.

The composition of a variety of raw cane and beet sugars is given in Table I.

TABLE I

Description of Sugar	Sucrose. Per Cent.	Glucose. Per Cent.	Organic Non-Sugar. Per Cent.	Ash. Per Cent.	Water. Per Cent.
Cane, Cuba (centrif.) ...	91.90	2.98	2.70	.72	1.70
Cane, Cuba (muscovado)	92.35	3.38	.66	.77	2.84
Cane, Jamaica	90.40	3.47	1.55	.36	4.22
Cane, Trinidad	88.00	5.14	1.67	.96	4.23
Cane, Porto Rico	87.50	4.84	2.60	.81	4.25
Cane, St. Vincent	92.50	3.61	2.45	.63	.81
Cane, Demarara	90.80	4.11	.77	1.12	3.20
Cane, Benares	94.50	2.63	.39	1.50	.98
Cane, unclayed Manila..	82.00	6.79	.24	2.00	5.97
Cane, concrete	84.20	8.45	1.70	1.10	4.55
Cane, melada	67.00	11.36	1.93	.91	18.80
Cane, bastards	68.30	15.00	1.20	1.50	14.00
Palm, East Indian	86.00	2.19	2.89	2.88	6.04
Beet, first	94.17		2.14	1.48	2.21
Beet second	91.68		2.49	2.92	2.91

66. Molasses and Cane-Sugar Sirups.—Molasses may be termed the mother liquor of the crystallized product—sugar. It is never found possible in practice, however, to crystallize all the sugar out and get a molasses that does not contain sucrose. The potassium salts and, in a lesser degree, the calcium salts that are present in the crude juice, are *melassigenic*, that is, prevent the crystallization of a certain amount of the sucrose; the invert sugar, or glucose, operates in the same way, and the long-continued heating of the sugar solutions also has the effect of increasing the molasses. In France, for instance, the *rendment*, or amount of crystallizable sugar obtainable in refining raw sugars, is calculated by deducting from the total sucrose twice the glucose and from three to five times the ash. In case of cane sugars the ash is not so melassigenic, not being so largely composed of potassium compounds as in the case of beet sugar, and a deduction of one and a half times the glucose is considered sufficient to allow for that impurity. The experience of the last few years with sorghum sugar, as manufactured by the United States Bureau of Agriculture and several sorghum-sugar factories in Kansas, has shown that this rule does not apply to sorghum.

With the sugar cane and sugar beet the percentage of sucrose, in both the raw molasses produced in the extraction of the sugar from the juice and the refined molasses, the sirup produced in the process of refining is quite large. The following analyses of a variety of molasses, shown in Table II, will illustrate the differences in the several grades.

TABLE II

Variety of Molasses	Sucrose. Per Cent.	Glucose. Per Cent.	Ash. Per Cent.	Organic Non-Sugar. Per Cent.	Water. Per Cent.
From Sugar Cane:					
Green sirup	62.7	8.0	1.0	.6	27.7
Golden sirup	39.6	33.0	2.5	2.8	22.7
Treacle	32.5	37.2	3.5	3.5	23.4
West Indian molasses	47.0	20.4	2.6	2.7	27.3
Dark molasses	35.0	10.0	5.0	10.0	20.0
From Beets:					
Beet-sugar molasses	46.7	.6	13.2	15.8	23.7
Beet-sugar molasses	50.0	none	10.0	20.0	20.0
Beet-sugar molasses	55.0	trace	12.0	13.0	20.0

It will be seen from the analyses that the percentage of sucrose is usually much higher in the beet-sugar molasses, a fact that is readily explained by the comparatively large amounts of ash and organic non-sugar. On the other hand, the invert sugar, or glucose, which takes a conspicuous place in the cane-sugar molasses, is nearly entirely absent in the beet-sugar molasses. The latter, however, always contains *raffinose*, another variety of sugar always present in the beet juice; *betaine*, a nitrogenous base; and *proteids*. The proportion of salts contained in beet molasses is usually from 10 to 14 per cent., whereas refiners' molasses from cane sugar rarely contains half that proportion.

67. The term **green sirup,** used in Table II, is given to sirup centrifugated from second products in the refining process.

Golden sirup is produced from refiners' molasses by diluting, filtering through bone black, and then concentrating.

Treacle is the name formerly given to the drainings from the dark-molasses sugars called bastards.

Cane-sugar molasses, when refined and brought to the condition of light-colored sirups, forms a common article of domestic consumption. The table sirups of the present day, however, as a rule, cannot claim to be simple products of the refining process, as they are almost always largely admixed with the cheaper glucose sirup, and the cane-sugar product in them is often entirely replaced by this latter. A glucose product, known as *mixing sirup*, is especially prepared and sold for this purpose.

Beet-sugar molasses is not adapted for use as table sirup, on account of the unpleasant taste and odor, due to the nitrogenous principles present. It is, as before described, worked for the extraction of the sugar, or it is fermented for alcohol.

68. Vinasse, or Molasses Residue. — When the beet molasses is fermented for the production of alcohol, the residual liquor, which contains all the potash salts of the molasses, is known in French as *vinasse* or in German as *schlempe*. Its specific gravity is approximately 41° Baumé and it has an acid reaction. It is neutralized with calcium carbonate, and then evaporated down to dryness and calcined. The black, porous residue so obtained contains from 30 to 35 per cent. of potassium carbonate, 18 to 20 per cent. of sodium carbonate, 18 to 22 per cent. of potassium chloride, 6 to 8 per cent. of potassium sulphate, and 15 to 18 per cent. of insoluble matter. It is exhausted with hot water, and the extract evaporated down, when potassium sulphate and, afterwards, sodium carbonate separate out. On cooling, potassium chloride and potassium sulphate crystallize out and the mother liquor contains potassium carbonate mixed with a small percentage of sodium carbonate. It is possible by this gradual evaporation and fractional crystallization to bring the crude potash to a purity of 90 per cent. In this production of the solid potash from the molasses residue, all the nitrogen of the molasses is lost. To prevent this, it has been prepared to submit the evaporated vinasse to a dry distillation instead of calcination in the air. The residue of this distillation is an open and very porous coke containing all the mineral salts of the molasses, which can then be extracted as before. The products of distillation are an illuminating and heating gas, ammonia water, and a small amount of tar. The ammonia water is the most interesting product. Besides carbonate, sulphide, and cyanide of ammonium, it contains methyl alcohol and notable quantities of trimethylamine. This latter compound can be decomposed at 320° C. by dry hydrochloric-acid gas into methyl chloride and ammonia, and on passing the products through aqueous hydrochloric acid, the methyl chloride goes through unabsorbed, while the ammonia is taken up. The methyl chloride is of considerable value for ice machines and for the manufacture of methylated aniline colors.

REFINING OF SUGAR

69. The processes previously described for the manufacture of raw sugar from cane or beet root yield in some cases sugar more or less fit for consumption, such as *beet crystals* and *Demarara*, but generally speaking, raw sugar contains impurities that are evident in its color, odor, and taste.

The process of refining is essentially the same for sugar of all sources, although, of course, a raw sugar of fair purity is treated more easily than one containing much foreign matter. Sugar, when completely refined, as in the case of loaf sugar of good quality, is almost pure sucrose, and is of identical flavor and sweetening power, whatever its origin. The idea that refined beet sugar is inferior in sweetness to cane sugar has arisen from the fact that formerly, when beet sugar was less completely refined than it is at present, its sweetness was diminished and its flavor impaired by the small quantity of alkali salts that it contained. Repeated tests have shown that it is now impossible to distinguish the source of refined sugar by taste.

Raw sugar varies considerably in composition, but a common content of sucrose is 90 to 95 per cent., the balance consisting of water, mineral constituents, organic matter other than sugar, and a small amount of invert sugar. The system of refining generally practiced is carried out in the stages described in the following articles:

70. Solution.—The raw sugar is dissolved in water by the aid of open or closed steam, a solution containing about 50 per cent. of sugar and having a temperature of about 70° C. being obtained. When very crude sugars are refined, defecation by the addition of blood, and heating until coagulation of the albuminous matter thus introduced occurs, is sometimes necessary.

71. Filtration.—The solution, if of beet sugar, may sometimes be filtered in a filter press; but if of cane sugar, it usually needs a different treatment. In this case, it is filtered through bags of cotton fabric (twill) arranged in an apparatus known as a *Taylor filter*. This filter consists of a cast-iron box covered with a perforated plate, from which depend long, narrow bags of hemp. Inside each bag is the filter bag proper, of twill, as stated above. The mouth of each bag is tied on to a nozzle set in one of the perforations of the plate covering the cast-iron chamber. This plate forms the bottom of a shallow tank resting on the chamber, and serves to distribute the sugar solution to the whole of the bags. By means of the large filtering surface exposed by the bags, filtration proceeds at a fair rate, notwithstanding the slimy character of the suspended matter in the sugar solution. The bags after use are either washed *in situ* or turned inside out and washed in tanks. In the former case, the weak liquor is used at once for dissolving a fresh batch of sugar, while in the latter case the turbid liquid is defecated with lime, filtered in a press, and then used for the same purpose.

72. Decolorization.—This is effected, as a rule, by animal charcoal. The charcoal is packed in large iron towers that are provided with perforated false bottoms and filtering cloths. The clear, warm sugar solution is run on to the charcoal at the top and the air expelled at the bottom until the tower is full, when it is allowed to stand for some time. The clear, decolorized liquid is then drawn off at the bottom of the tower, while fresh liquid is run on at the top, the operation being continued until the liquid ceases to be satisfactorily decolorized. When the yellow color of the liquid shows that the decolorizing power of the charcoal has been exhausted, the supply of fresh liquor is cut off and a stream of water is substituted. A great deal of liquor still remaining in the tower is thus expelled without being much diluted by the incoming water, but the remainder, known as *char water*, is comparatively poor in sugar and is returned to the cycle of operations—e. g., used for dissolving the raw sugar. The function of the char consists in the withdrawal from the sugar solution of many impurities, both organic and inorganic. The rational action of the animal charcoal is not well understood, but it has been found that wood charcoal, although generally richer in carbon, cannot be substituted. The fine state of division of the carbon in the animal charcoal is probably one cause of its efficacy, but its content of matter other than carbon, notably nitrogen, also appears to be concerned in its decolorization influence.

73. Revivification of Charcoal.—By revivification of the charcoal is meant the separation from it of those saccharine and other matters that it absorbs in the clarification process, making it fit to use again. With this object in view, it is washed and reburned. The washing is generally effected by prolonged treatment of the char with boiling water; but sometimes hydrochloric acid is used in amount calculated to destroy the calcium salts taken up from the sugar solution, while leaving those naturally present in the original char unattacked. The organic matter may be also eliminated by allowing fermentative decomposition to set in, and then washing out the products of the changes thus produced. The washing, however performed, is followed by *reburning*, the process consisting in heating the char, out of contact with air, to a temperature of about 640° to 650° C. A pipe kiln is generally adopted for reburning, though revolving cylinders, externally fired, are sometimes used. In the case of the former, the char is fed into the upper ends of cast-iron pipes of elliptical section, set vertically in groups, and heated from below by a furnace. The wet char thus enters at the cooler ends of the pipes, is there dried, and is more strongly heated as it descends, finally emerging at the lower and hotter end of the pipes into sheet-iron cooling pipes provided with valves, so that successive portions of the reburned char can be dropped out and conveyed to the decolorizing towers. After repeated burning, the char ceases to act efficiently, and it is disposed of for fertilizing purposes.

74. The practical test to determine whether the revivification has been properly conducted is qualitative, and for which a caustic-soda solution is used as follows:

Boil about 50 grams of bone black 2 or 3 minutes in 50 cubic centimeters of a solution of caustic soda (9° Brix, or 5° Baumé). Decant, or filter off, the solution into a test tube and note its color. A faint tinge of color indicates a good revivification; a colorless or greenish solution indicates over-revivification. This test is of great importance, and should be made frequently. A reddish-tinged char indicates imperfect revivification; gray, leakage of air in the retorts; and white, an overburned bone black. A greenish color on treatment with caustic soda is an indication of the presence of sulphide of calcium. Occasional tests should be made for this substance, since the same affects the quality of the bone black, and when present in more than very small quantities, the char should be rejected. This salt may be tested for qualitatively by treating the char with strong acid and testing the gas liberated for hydrogen sulphide H_2S.

75. Weight of a Cubic Foot of Bone Black.—Bone black naturally increases in weight each time it is used, by the absorption of impurities that are not removed in the revivification. This gradual increase in weight is a measure of the deterioration from usage. It is impossible to give a standard weight of bone black, as this depends largely on the size of the grains; but it has been shown that the weight of 1 cubic foot ranges from 43 to 48 pounds for new char. On commencing work with new char, its weight per cubic foot should be taken and recorded and this weight used for future comparison. According to good authorities, the weight of good bone black while in use should increase more than 23 per cent. when new; at an increase of 47 per cent. it is very bad; and an increase of 50 per cent. makes it useless for further use.

76. Decolorizing Power of Bone Black.—The decolorizing power of bone black is determined by means of a colorimeter. Stammer's instrument is very convenient for this purpose, and the results obtained by careful operators compare very closely. This instrument consists essentially of an arrangement for comparing the depth of color of a column of sugar solution with standard colored-glass plates. An eyepiece is so arranged that the color of the solution under examination appears upon one-half of the disk and that of the standard glass on the other. The eyepiece and tube containing the glasses are raised and lowered by means of a rack and pinion, the length of solution being varied at the same time; this length is shown on a scale by means of a pointer carried by a slide. The theory of this instrument depends on the variations in the depth of the color of the solution, proportionate to the length of its column. In using the colorimeter, the object is to equalize the intensities of the colors, as seen on the disk through the eyepiece, by lengthening or shortening the column of the solution under examination. The strength of the solution being known, a comparative statement of depth of color in terms of the sucrose present may be made, or the reading on the scale may be easily reduced to an expression showing the depth of color as compared with the standard.

This instrument may be used in determining the decolorization power of a char in the following manner: A standard color solution is prepared by using a definite quantity of caramel—Dubosq recommends 2 grams per liter. Prepare the caramel by heating pure cane sugar to about 215° C. until all the sugar is decomposed. In examining bone black, determine the depth of color in the standard solution, then heat a measured volume of this solution with a weighed portion of the char a certain length of time—for instance, 30 minutes—filter, and again determine the intensity of color. The difference in the depth of color referred to the standard represents the efficiency of the bone black in decolorizing. In sugar-house work, a standard bone black of a known decolorizing capacity is convenient for comparison. Comparable results can only be obtained by adopting certain conditions and adhering to them in all experiments.

The decolorizing power may be roughly determined, in the absence of a colorimeter, as follows: Treat a measured volume of a standard color solution as described before. Fill a graduated cylinder, similar to those used for the colorimetric determination of carbon in steel, to a certain point with the decolorized and filtered solution, take the same volume of the standard solution in a similar cylinder, and add water to this latter from a burette until a portion of the same depth as that of the decolorized solution shows the same intensity of color when examined over a background. The volume of water added is inversely proportional to the decolorizing power of the char.

77. Evaporation of the Decolorized Liquor.—The liquor is boiled down in a vacuum pan similar to that used in concentrating purified juice. The boiling down is carried on at a temperature ranging from 49° to 88° C., according to the quality of the sugar to be produced. In general, the larger the size of the crystals required, the higher the temperature adopted. The advantages of the use of vacuum pans have been already mentioned.

78. Sampling Sugars and Molasses. — Too much importance cannot be attached to the securing of correct samples, that is to say, to obtain samples that shall be representative of the substance examined.

The samples of raw sugar are drawn with a long steel bar resembling the half of a pipe cut longitudinally. A hole having been made in the package, the *trier*, as it is called, is inserted, rotated completely, and then withdrawn. The sample that fills the hollow in the trier is removed and is placed in a can. When sirup or molasses is to be sampled, a rod or a stick is inserted in the bung hole of the barrel and rapidly withdrawn; the adhering liquid is placed in a can and the operation repeated until sufficient has been obtained. When sugars in hogsheads are sampled, the hogshead is placed on its side. The manner of inserting the trier differs. The government takes its sample by running straight through the contents from center to center of the heads; at some refineries the trier is run through diagonally from head to head. Meladas are sampled through the bung hole of the hogshead. In a refinery, all sugars, sirups, and molasses are sampled.

The United States Government varies its requirements as to the number of packages to be sampled, with the nature of the package: Of hogsheads, tierces, boxes, and barrels, 25 per cent. are required for sample and 100 per cent. for a resample; of centrifugals and of beet sugars, in bags, 5 per cent. for sample and 5 per cent. for resample; of mats, 2½ per cent. for sample and 2½ per cent. for resample; of baskets, 10 per cent. for sample and 10 per cent. for resample; of "Joggeries," Pernambuco, and Brazil sugars, 5 per cent. for sample and 5 per cent. for resample.

When the samples have been taken and are brought to the laboratory for analysis, it is necessary either to make a separate analysis of every mark in a lot, or, as this is generally not feasible, to prepare a representative sample. In order to do this, fix upon some definite quantity, by weight, as the unit weight. Weigh out this amount, proportionate to the number of hogsheads in each mark, and place in a well-closed jar. For example, suppose a lot of sugar contained four marks, A, B, C, and D.

Mark A = 1,000 hogsheads
Mark B = 200 hogsheads
Mark C = 350 hogsheads
Mark D = 70 hogsheads

Then take from:

A = 100 grams
B = 20 grams
C = 35 grams
D = 7 grams

For analysis, if necessary, crush the sample, thoroughly mix the contents of the jar, and then proceed as usual. As some lots come in mixed packages, that is to say, partially in hogsheads, bags, tierces, and barrels, a certain relation between these has been assumed; it is as follows:

1 hogshead = 2 tierces
1 hogshead = 8 barrels
1 hogshead = 8 bags

To prepare average samples of refined sugars, proceed as directed above.

79. Determination of Color of Sugar and Sugar Solutions.—The color tests made on sugars and on sugar solutions are generally only comparative—that is to say, the color of the sample examined is compared with that of some other sample which is taken as the standard.

In the examination for color of raw sugar, the so-called *Dutch standards* are usually employed. These consist in fifteen samples of raw sugar, numbered from No. 6 to No. 20, and ranging in color from a dark brown (No. 6) to almost a white (No. 20). They are prepared and sealed with great care by a certain firm in Holland. The samples are renewed every year, and serve as standards for the 12 months following their issue.

80. In examining the color of sugar solutions, to learn, for instance, how effectively a certain sugar has been decolorized in passing through bone black, two test tubes, beakers, or cylinders made of colorless glass are filled to an equal height with, respectively, the sample under examination and the *standard* solution with which the sample is to be compared, both solutions of course being of equal density.

Various forms of apparatus have been designed for effecting color comparison. In some, the standard solution is replaced by colored-glass disks of tints ranging from a pure white to a dark yellow or brown; by combination of these it is possible to produce almost any shade desired. The colorimeter probably most used is that of Stammer. As the depth of color of a solution is proportional to the length of a column of such solution, there is ascertained in this instrument the height of a column of the solution that will, in color, correspond to the tint of a standard colored-glass disk inserted in an adjoining tube. The scale is graduated in millimeters. If, for instance, a depth of 1 millimeter of the solution corresponds to the normal tint, the color is said to be 100. If 2 millimeters depth of solution are required to match the tint, the color is 50; if 4 millimeters, 25; and so on.

PETROLEUM AND PRODUCTS

(PART 1)

NATURE AND COMPOSITION OF PETROLEUM

1. Petroleum, also known as rock oil, maltha, and Seneca oil, is one of nature's products that is widely divided over the surface of the earth. It has been found in springs, oozing from the ground, in shallow wells, and Artesian wells many thousand feet deep. In this country, it is found in one or the other of its various forms in almost every state, although it seems to occur in paying quantities in comparatively few of them.

It is found in Pennsylvania, New York, and West Virginia, in sandstone of various geological formations, which is found in several distinct layers, known as the first, second, third, fourth, and fifth oil sands. We thus find wells of various depths; while a well, say 60 or 70 feet deep, may be drilled to the first sand and produce oil, another, perhaps, must be drilled to the third or fourth sand before oil is found and may be 600 feet or more deep.

2. Chemical Composition of Petroleum.—Petroleum is composed in varying quantities of almost the entire series of pure hydrocarbons known. In American petroleum, the methane or C_nH_{2n+2} series seem to predominate. Sadtler found natural gas, which is now generally acknowledged to belong to the same class and have the same origin as petroleum, to be composed almost entirely of methane, ethane,

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and propane. The first, or marsh gas, constituted about three-fourths of all the gas analyzed. Peckham gives the following analysis of a number of noted American gas wells.

Well	CO_2	CO	H	CH_4	C_2H_6	C_3H_8
Burns	.34	trace	6.10	75.44	18.12	trace
Leechburg	.35	.26	.56	89.65	4.39	trace
Harvey	.66	trace	13.50	80.11	5.72	trace

Burns's well is in the northern part of Butler County, Pennsylvania; Leechburg is in Armstrong County, and the Harvey well is in the southern part of Butler County. Nitrogen has been found in the gas from the Baku fields, but very little has been reported to exist in any of the Pennsylvania gas.

3. The first chemical examination of the liquid products of the wells, that seems to concern the petroleum industry, was that of Professor Benjamin Silliman, Jr., in 1855. In the earlier period of the industry it was assumed that petroleums from different localities were identical, except in specific gravity and that, therefore, the distillate of the same specific gravity possessed the same properties. Chemical analysis has shown that this by no means is the case.

4. While the methane, or marsh gas, series predominates in America, the naphthene, or more condensed series, predominates in Russia. This gives rise to a marked difference in the general properties of the products obtained from these crude oils. Mendeleeff considers that although the two crude oils contain almost the same hydrocarbons, the proportion is far from being the same. From a large number of Baku crude oils examined by him, he found that the specific gravity of the distillate, obtained between 100° and 105° C., after four or five fractionations varied between .701 and .756; while the corresponding fraction of American petroleum lies between .703 and .71.

TABLE I

HYDROCARBONS, C_nH_{2n+2}, ISOLATED FROM AMERICAN CRUDE OIL

Name	Chemical Formulas	*C*	*H*	Boiling Point	Specific Gravity	Remarks
GASEOUS						
Methane	CH_4	75.00	25.00		.559	
Ethane	C_2H_6	80.00	20.00		.5516	
Propane	C_3H_8	81.81	18.19	−20° C.	1.522	Colorless liquid at −20° C.
Butane	C_4H_{10}	82.80	17.20	1°	.600 (0° C.)	Colorless liquid of sweet taste
LIQUID						
Pentane	C_5H_{12}	83.33	16.67	37°	.628	
Hexane	C_6H_{14}	83.72	16.28	69°	.664	
Heptane	C_7H_{16}	84.00	16.00	9~~7~~8.5°	.699	
Octane	C_8H_{18}	84.21	15.79	125°	.703	
Nonane	C_9H_{20}	84.38	15.62	136°	.741	
Decane	$C_{10}H_{22}$	84.51	15.49	158°	.757	Flash 26° C., fire 38° C.
Undecane	$C_{11}H_{24}$	84.61	15.39	182°	.765	Flash 40° C., fire 58° C.
Dodecane	$C_{12}H_{26}$	84.70	15.30	198°	.776	
Tridecane	$C_{13}H_{28}$	84.78	15.22	216°	.792	
Tetradecane	$C_{14}H_{30}$	84.85	15.15	238°	.812	
Pentadecane	$C_{15}H_{32}$	84.90	15.10	258°	.825	
Hexadecane	$C_{16}H_{34}$	84.94	15.06	280°	.828	
SOLID						
Paraffin myricyl	$C_{27}H_{56}$	85.26	14.74			
Paraffin ceryl	$C_{30}H_{62}$	85.31	14.69	370°		

5. We are indebted to Schorlemmer, Pelouze, Cahours, and Warren for our knowledge of the composition of petroleum. The compounds of the methane series given in Table I have been isolated from Pennsylvania petroleum and examined.

6. Besides the methane series, given in Table I, isomers of a great many of these hydrocarbons, having the same composition and specific gravity but a slightly different boiling point, have been isolated and identified. Höfer has isolated a series of olefines; Chandler has found benzene and some of its homologues in the Bradford crude; and Peckham gives an extended account of the discovery of petrocene and thallene in the tar distillation of petroleum. A number of other compounds have been mentioned as existing in small quantities; but since it is impossible to find many of these compounds without distilling large quantities of the crude oil, it is quite likely that many of them are simply products of condensation caused by the heat of the still.

7. The relative proportions of these different compounds existing in petroleum have never been fully determined. Yet an approximate idea may be obtained by distilling Pennsylvania petroleum and referring the results to Tables I and II. The latter table gives the commercial products and the compounds supposed to be contained therein.

8. Oxygen is shown to exist in nearly all petroleum, but much of this may be due to absorption, since all distillates are known to absorb it quite freely. Nearly all the oxygen is contained in the form of acids and phenylated compounds.

Table III, which was prepared by Aisinman, gives the elementary composition of petroleum from various localities.

9. Besides the three elements given in Table III, nitrogen has been found in nearly all crude oils. This element is usually present in the form of bases. Peckham gives it as his opinion that it exists not only as pyridine and quinoline, but that a large number, if not all, of the methylated compounds of these bases are associated with them.

TABLE II

PRODUCTS MANUFACTURED FROM PETROLEUM

Name	Boiling Point. Degrees Centigrade	Specific Gravity	Use
Natural gas	Gas		Fuel
Rhigolene	0°	.6222	Local anæsthetic
88° Gasoline	50°	.6400	Extraction of oils and air-gas machines
80° Gasoline	70°	.6660	Air-gas machines
Stove gasoline	98°	.7000	Fuel in a special stove
63° Deodorized	110°	.7250	Substitute for turpentine
Kerosene	150°	.7860	Illuminant
Lubricating oils		.8750– .9150	Lubricants
Vaseline		.8917– .9333	As a vehicle for salves
Paraffin			Candles and insulation

10. The element that has been of especial annoyance to the American refiner is sulphur. This is found in Ohio, Indiana, and Canadian oils in such quantities as to give them a marked odor that increases during distillation. Mabery has made an extended examination of these oils, and reports the percentage of sulphur in the Ohio and Indiana crudes never to exceed .6 per cent. He states that these compounds are sulphides of methyl, ethyl, normal propyl, normal and isobutyl, pentyl, ethyl pentyl, butyl pentyl, and hexyl, but that neither mercaptan nor thiophene were found. In Canadian petroleum he found .98 per cent. of sulphur, besides a quantity present as hydrogen sulphide.

11. Inorganic substances are always found in petroleum, but only in minute quantities. Silver, copper, mercury, iron, arsenic, and even gold have been found in the ash of

TABLE III

Locality	Sp. Gr. at 0° C.	Elementary Composition			Remarks
		C	*H*	*O* by Difference	
Baku	.882	87.40	12.60	.10	It is composed of naphthenes and a few other hydrocarbons, as well as oxygen and sulphur compounds; in the lighter fractions small quantities of paraffin are found.
Baku	.884	86.30	13.60	.10	
Baku	.897	86.50	12.00	1.50	
Baku	.928	87.10	11.70	1.20	
Baku, very heavy	.938	86.60	12.30	1.10	
Baku	.954	85.30	11.60	3.10	
Baku		86.00	13.00	2.00	
Pennsylvania, near Franklin	.886	84.90	13.70	1.40	The Pennsylvania crude oil consists principally of the paraffins, with a small percentage of the aromatic series and a minute quantity of the naphthenes in the heavy distillates. All American petroleum contains oxygen and sulphur compounds and a few, also, nitrogen compounds.
Oil Creek	.816	82.00	14.80	3.20	
W. Canada	.857	81.30	13.40	2.30	
Petrolia	.870	84.50	13.50	2.00	
West Virginia	.841	84.30	14.10	1.60	
West Virginia	.857	83.20	13.20	3.60	
West Virginia	.897	83.60	12.90	3.50	
Kentucky		85.20	13.36	1.11	
California		86.93	11.82	2.70	
California		86.62	12.92	.54	
Ohio	.887	84.20	13.10	.70	
Ohio, Mecca		86.30	13.07		
West Galicia	.855	85.30	12.60	2.10	A mixture of paraffins, naphthenes, and aromatic hydrocarbons.
East Galicia	.870	82.20	12.10	5.70	

TABLE III—*Continued*

Locality	Sp. Gr at 0° C.	Elementary Composition			Compounds Found in Petroleum
		C	H	O by Difference	
Roumania	.901	83.00	12.20	4.80	
Roumania	.770	82.60	12.50	4.90	
Italy, Parma	.786	84 00	13.40	1.80	
Italy, Piedmont	.919	86.40	12.20	1.40	
Germany, Hanover	.892	80.40	12.70	6.90	Principally paraffins, contains also aromatic hydrocarbons and naphthenes.
Germany, Oberg	.944	84.40	11.50	4.10	
Germany, Witze	.955	86.20	11.40	2.40	
Germany, Alsace	.912	86.90	11.80	1.30	
Germany, Pechelbronn	.968	85.60	9.60	4.60	
Germany, Pechelbronn	.892	85.70	12.00	2.30	
Germany, Pechelbronn		86.10	12.70	1.20	
Germany, Pechelbronn		83.30	11.10	1.10	
Germany, Schwabweiler	.861	86.20	13.30	.50	
Germany, Schwabweiler	.829	79.50	13.60	6.90	
Germany, Schwabweiler	.820	85.50	14.20	.30	
East India, Burmah	.875	83.80	12.70	3.50	
Java, Rembang	.923	87.10	12.00	.90	
Java, Tjabados, Fanga	.827	83.60	14.00	2.40	
Java, Gagor	.927	85.00	11.20	2.80	
China, Fu-li-fu	.860	83.50	12.90	3.60	

petroleum. These elements seem to have but little effect on the technology of petroleum, which is affected more by the varying quantities of light and heavy hydrocarbon compounds that exist in petroleum than by almost any other factor in the composition.

TECHNO-CHEMICAL EXAMINATION

12. This examination is of much greater importance to the manufacturer than any ultimate analysis. Under this head will first be considered such physical properties as color and specific gravity. The color of the American crude petroleum varies from a clear water-white, through a straw-yellow, light amber, red, deep red, dark brown, to an opaque black.

The first, although only a freak in the petroleum fields, has been found in a well at Holders Run, Armstrong County, Pennsylvania. The others are of common occurrence and may be seen in the crude oils of West Virginia, Pennsylvania, Ohio, Indiana, California, and Wyoming, respectively. Each of these States has produced crude oils of various shades, the light-colored oils usually coming from some strata nearer the surface than those of a darker shade. Table IV, taken from Aisinman's Taschenbuch, gives these physical properties for petroleums from several localities.

13. Nearly all crude oils have a fluorescence varying from blue to dark green. That of Pennsylvania is of a beautiful light grass green. The specific gravity of crude oil varies from .77 to .98, all being lighter than water. By referring to Table IV, it will be noticed that there exists a certain connection between color and specific gravity, the lighter colored oils usually having a lighter specific gravity, and vice versa.

14. The variability mentioned in the preceding articles depends partly on the chemical composition and partly on the physical condition of the oil. All crude oils hold more or less natural gas in solution, which lowers their specific gravity; on the other hand, oils containing a comparatively large percentage of paraffin or asphalt have a greater specific gravity—this also applies to all sulphur oil. It has been further found that oils having a low specific gravity furnish a greater percentage of gasolines and illuminating oils than those that are heavier; the specific gravity of an oil plays an important part in the price of the oil.

TABLE IV

COMPARISON OF COLOR AND SPECIFIC GRAVITY OF CRUDE OIL

Specific Gravity	Color	Odor	Locality
.777	Light straw	Strong, unpleasant	Persia
.787	Light straw	Pleasant	Near Milan, Italy
.810	Light amber	Pleasant	Bradford, Pennsylvania
.818	Light amber	Faint, pleasant	Mud volcano, Burmah
.828	Light brown	Very faint	New Zealand
.829	Amber	Unpleasant	India
.835	Dark brown	Faint, pleasant	East Borongo
.836	Dark reddish brown	Faint, pleasant	Russia
.843	Dark brown	Faint, pleasant	Hanover
.852	Dark reddish brown	Faint	South America
.865	Dark brown	Penetrating	Canada
.866	Dark brown	Faint, pleasant	Burmah
.888	Chestnut brown	Faint, pleasant	Burmah
.900	Dark brown	Very faint, somewhat viscous	South America
.910	Black	Unpleasant	Wyoming
.913	Brown-black	Unpleasant	Hanover
.933	Dark brown	Faint, pleasant, viscous	Assam
.935	Dark brown	Viscous	India
.942	Brown-black	Viscous	Russia
.945	Black	Unpleasant	Wyoming
.957	Black	Tar	Barbadoes, C. A.

The more important factor in setting the price of oil, however, is its chemical composition and freedom from suspended matter. Heavy oils hold water and earthy matter in suspension and must be heated in order to settle them before they are received by the pipe line. This causes a loss of the more volatile parts and necessarily makes them less valuable. The sulphur oils, besides being heavier, require a more expensive treatment in order to produce a first-class illuminating oil and, consequently, are not so desirable to the refiner.

When an oil contains very small percentages of gasoline and burning oil, and, in consequence, has a high fire test and is free from sediment and wax, it may be of a much higher value as a lubricant, requiring no refining. Such oils are sold as natural lubricating oils.

15. The refiner generally attempts to produce all, or a part, of the compounds given in Table II by fractional distillation and subsequent purification with sulphuric acid, caustic soda, and such other chemicals as the distillate may require.

The specific gravity being carefully taken, a given quantity of the crude oil is heated to 120° F. and poured into a graduated cylinder to settle. After standing for 10 or 12 hours, the amount and nature of the sediment is determined. Another sample of definite quantity of the crude is weighed and then dried for 24 hours over sticks of chloride of calcium. The difference in weight is ascribed to the loss of water, from which the percentage of water can be readily calculated.

The presence of certain offensive sulphur compounds may be detected by the odor, but they will be sure to appear in the distillates. The condition of these compounds is best determined by treating the distillate with a small quantity of sulphuric acid, decanting, washing the oil with water, neutralizing with caustic soda, and testing the sample with sodium plumbate, as described later.

16. Fractional Distillation Test.—For the fractional distillation test, a flask of about 150 cubic centimeters capacity, having an outlet tube about 7 centimeters above the bulb and extending downwards at an angle of 75°, as shown in Fig. 1, is used. The observance of exactly these dimensions is essential in order to obtain uniform results. 100 cubic centimeters of previously dried crude oil is introduced and a thermometer fixed in the cork inserted in the mouth of the flask, so that the top of the bulb is on a level with the vapor-outlet tube; the vapor tube is connected with a Liebig condenser, and an Erlenmeyer flask is used as a receiver.

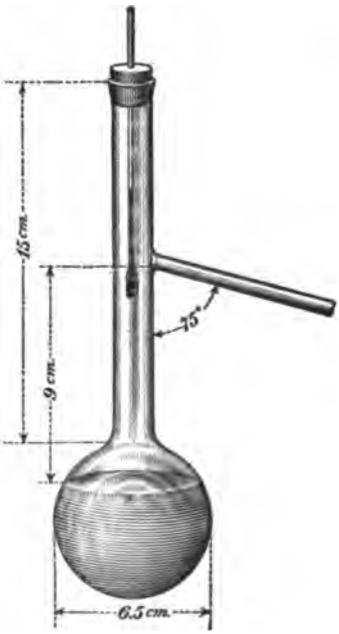

FIG. 1

17. The flask and contents are gradually heated with a Bunsen gas lamp; it is well to interpose a wire gauze between the flame and the flask in the beginning, but afterwards this should be removed and the naked flame employed. The heat is so regulated that from 2 to 2.5 cubic centimeters of distillate passes over per minute. The fractions usually taken are: up to 150° C., naphtha; 150° to 300° C., burning oils; over 300° C., residuum.

As soon as the thermometer indicates the temperature at which it is desired to cut off the fraction and begin the next, the temperature is permitted to drop 20° and then reheated to the original temperature. This is done as often as any appreciable quantity of oil is obtained before the fractionating temperature is reached. The first fraction having been completed, the second is taken in the same way. All over 300° C. is permitted to remain in the still, or it may be further divided into a secondary oil and a tar of 19° to 21° Baumé.

In the same manner, closer fractions may also be taken, as at each 50° C.; but in practice the manufacturer only desires to know the fractions given above. The results will not agree entirely with those to be obtained on a larger scale, but are sufficiently accurate to give a comparative idea of the crude oil under consideration. The distillates should now be tested as to gravity and general purity.

18. Many practical refiners look upon such results as inaccurate. It must only be considered comparative and the refiners' business must determine what percentages may be obtained. In most cases, the refiner is able to increase his output of illuminating oil as much as 25 per cent. over the laboratory figures. If the operator in the laboratory, however, will observe the products distilling over above the 300°C. (572° F.), he will usually find that considerable oil in this fraction may be used with the illuminating fraction without injury to the color, though they are of much higher gravity.

19. The different States have different requirements, and if the sales department handles the products to the best advantage, it will be able to dispose of a large percentage of its heavy oils as illuminants of low fire test, or as high-fire-test railroad or lighthouse oils. The former may be accomplished by mixing with the heavy oil a fraction of the heavy naphtha distilling over just before the 150° C. cut. This produces a good burning oil, but low in fire test. If, however, the refiner wishes to sell "gilt-edged" illuminating oils of high gravity and fire test, he must dispose of his heavy oils in some other way. A low-fire-test illuminating oil is usually a better burning oil than one of high fire test, and without doubt many of our States have acted unwisely in placing the fire-test requirement so high (see State's requirements given later). Most foreign markets have a low-fire-test requirement, and this has permitted some refiners to produce as much as 80 per cent. of illuminating oil from Pennsylvania crude oil.

20. The tests given in Table V will be found accurate in the production of a good burning oil of 150° F. fire test.

21. In making a careful examination of the distillation tests given in Table V, it will be observed that the petroleum

TABLE V

Name of Oil	District From Which Sample Was Taken	Specific Gravity. Degrees Baumé	Boiling Point. Degrees Centigrade	First Fraction		Second Fraction		Third Fraction		Fourth Fraction		Sulphur	Water	Sediment
				Per Cent. by Volume up to 150° C.	Gravity of Distillate. Degrees Baumé	Per Cent. by Volume. 150° to 300° C.	Gravity of Distillate. Degrees Baumé	Per Cent. by Volume, from 300° C. Upwards	Gravity of Distillate. Degrees Baumé	Per Cent. by Volume of Residuum	Gravity of Residuum. Degrees Baumé			
Pennsylvania	Emlenton, Pa., July 10, 1900	46.5	80	18.2	67.0	32.45	44.3	36.50	34.5	11.42	19.3			
Scio	Scio, Ohio, April 15, 1899	48.1	40	23.33	69.9	30.00	47.0	40.23	42.5	6.16	19.5			
Barnesville	Barnesville, April 15, 1900	46.3	40	24.50	70.0	41.90	45.6	22.60	39.7	10.00	20.0	*		
North Lima	Tiffin, Ohio, April 30, 1898	42.1	40	21.87	67.0	43.75	44.4	19.53	34.7	19.75	20.8	*		
North Lima	Freeport, Ohio, April 30, 1898	41.0	70	17.60	60.0	41.60	44.3	17.70	39.0	22.30	19.7	*	½%	
North Lima	E. Toledo, Ohio, May 14, 1898	35.0	80	10.71	59.0	31.25	44.3	29.35	38.5	22.67	19.3	*	3%	*
Indiana	Warren, Ind., June 10, 1898	33.0	83	4.00	65.0	28.50	45.0			67.00	25.3	*	5%	*
Texas	Corsicana, June 14, 1898	38.5	55	11.20	67.2	49.63	45.6	13.98	33.5	23.75	19.3			
Kansas		32.0	60	14.25	58.0	78.20				2.50	17.9			
California	Ventura County	28.0	60	9.70	60.0	29.10	40.0			61.20	17.0			
California	Fresno County	36.0		33.06	49.0	57.85	30.0			9.09	19.5	*		
Baku		32.0		8.50		40.70		18.30		20.00				
Baku	32% lubricating oil	28.0				26.00		10.00		10.00				
Baku		33.0		23.00		38.00				39.00				

* Indefinite quantities.

obtained from the different fields yields varying quantities of light and heavy oils. The first eight samples were carefully selected and all tested in precisely the same manner, so as to yield concordant results. The tests were all made for the purpose of giving certain manufacturers some idea of the value of these oils when distilled for commercial purposes. The other samples were taken by different authors; and doubtless there was some difference in the method of testing. The second and fifth samples have been distilled and refined on a commercial scale in the Sun Oil Company's refinery, when the same results were obtained.

22. In order to understand the value of such tests, let the Pennsylvania oil be taken as a basis and compare the other oils with it. In this way it will be seen how the practical refiner will be able to make a choice. The first three yield very nearly the same results and are, at present, rated under the head of Pennsylvania oil in the American crude-oil markets. The second and third were taken from new territory, and consequently yielded a large percentage of light oils. The second gave exceptionally fine "first" and "second-grade" oils of high gravity, and on that account received a premium in advance of the market price at the time these tests were made. Since the increasing demand for petroleum cylinder oils, the oils that are free from sulphur and have a light color and no sediment are manufactured into cylinder stocks. The Barnesville oil being of a darker color and not perfectly clear will not produce as fine a cylinder stock, and is, therefore, not valued as highly; but since the Pennsylvania crude stocks are decreasing, this oil enters the pipe lines and, being mixed with the other oils, passes unnoticed. The fourth oil given in Table V is a darker oil and contains an objectionable amount of sulphur.

23. In looking over the percentage columns of Table V, but little difference is found between the first North Lima oil and the preceding ones, excepting that it yields a larger percentage of residuum. The Tiffin field produced some of the finest North Lima oil thus far obtained from the State of Ohio. As will be observed, its gravity is quite good; but since it contains sulphur in such combination which has been found difficult to remove, it is not of a high value. With the increasing demand for kerosene, paraffin, and other petroleum products, the processes for refining this oil have been constantly improved until the refiner is able to place it on the market in competition with products of the higher priced petroleum.

The prices of the Pennsylvania and Ohio oils have been gradually approaching each other and, doubtless, within a a very few years an oil similar to that from Tiffin will command the same price as that from the older fields in Pennsylvania. The fifth sample contains less light naphthas and more heavy oils.

The sixth and seventh samples are much poorer grades, being of little value to the refiner; the moisture and sediment are particularly objectionable. The principal use for oils of this kind is as fuel.

24. The Texas oil is comparatively free from sulphur, and is finding considerable use as crude oil to be refined. Its products are quite good, with the exception of the presence of asphalt.

The California oils have been examined of late by Professor Mabery. His reports show nitrogen compounds and considerable heavy oils. His vacuum distillations have brought out a fine line of lubricating oils almost entirely free from crystalline paraffin. Developments are now proceeding so rapidly in this field that these analyses are by no means sufficient to form a judgment of the crude oil of this State. The greater part of it is still finding its market as fuel.

25. In comparing the Baku petroleum with the other samples, it is necessary to take into consideration the fact that the oil is principally composed of the naphthenes, which are much less volatile than the compounds of the American petroleum. Redwood states that as much as 50 or 60 per cent. of the oil sold is residuum.

NATURAL GAS

26. Occurrence.—The history and development of the **natural gas industry** are so closely allied with that of petroleum as to be practically a part of it. As Doctor Orton has said, "They have a common history, produced from the same sources, accumulated by similar agencies, and stored in the same reservoirs. . . . Petroleum never exists free from gas, but it is sometimes asserted that gas is found that has no connection with petroleum. This claim is doubtful, and if the driest gas could be followed throughout its underground reservoirs, it is altogether probable that accumulations of oil would be found in every case."

27. The knowledge and use of natural gas in America antedates that of oil. **Burning springs,** as they were called, were observed by the earliest settlers. Nearly all of the salt wells, besides containing oil, produced some gas; the gas from the salt wells along the Muskingum was utilized in evaporating the brine. It was encountered in drilling the early oil wells, and was looked upon as a nuisance.

28. Uses of Natural Gas.—Fredonia, New York, has the distinction of being the first town to use natural gas as an illuminant; in 1824, it was used to illuminate a mill and a number of houses in the town. During the early operations on Oil Creek, in 1862, it was used as fuel in firing the boilers in pumping wells.

In 1867, it was piped to some houses in Oil City, where we have the first record of its being used for cooking and heating dwellings. The first attempts were not considered very successful on account of the changing pressure, but this objection having been overcome, it was regularly installed for such purposes. It has since held the distinction of being the most convenient and economical fuel to be had for domestic use. The cities and towns about the oil fields were quick in adopting its use. It was also found to be valuable in the furnaces of iron mills and glass factories.

29. When the "oil fever" had taken possession of the people throughout the country, on account of the successes on Oil Creek, the earth was punctured in almost every quarter of the United States, and it was soon observed that gas was more widely distributed than oil.

30. The celebrated Harvey well, in the southern part of Butler County, Pennsylvania, was struck in November, 1874, at a depth of 1,145 feet. It was located between abrupt hills in a valley about 300 feet wide. The gas was conveyed a distance of 150 feet in a 6-inch iron pipe, from which it discharged with the force of steam. The gas was allowed to burn for some time, as it escaped in a flame 40 feet high and 15 feet wide.

In the Fall of 1875, Spang and Chalfant piped it to their rolling mill at Sharpsburg, a distance of 17 miles. The Burns well on the Duffy farm, near St. Joe, in the same county, was struck in 1875 at a depth of 1,600 feet. The output of this well averaged 12,000,000 cubic feet of gas per day. It was piped to Freeport, where it was used for fuel and illuminating purposes. For heating purposes it was considered 25 per cent. better than bituminous coal.

The Delamater well in the same county was an oil producer so long as the owners did not go below the third sand. When they did, they lost a 10-barrel well and struck what appeared to be an inexhaustible reservoir of gas, although the volume of gas was not so large as that of the Burns well, one-half mile distant. This well was also connected with the line leading to the Sharpsburg rolling mills.

Although the manufacturers of Pittsburg knew of the value of the gas in the oil regions, they did not make any use of it for 20 years or more. But finding that a better quality of iron could be produced at about two-thirds the cost, it was then readily adopted. Glass factories, also, began to use it, and found that a better quality of plate glass could be made.

In 1877, the Haymaker well opened the Murraysville field, and was piped to Pittsburg by Pew and Emerson. The Philadelphia Gas Company procured large tracts of land and sent their product to Pittsburg.

At East Liverpool, Ohio, the gas was used as a substitute for coal in the early seventies. It has been one of the causes for the building up of one of the most prosperous pottery industries of America.

31. The discovery of high-pressure gas in the Trenton limestone belongs largely to Dr. Charles Oesterling, of Findlay, Ohio. Natural gas had been known in Findlay since the country was first settled. In digging wells, cisterns, and sewers, in springs and rock crevices inflammable gas had been found. It had been utilized in a small way for a number of years. Gas was first used there as an illuminant by Daniel Foster, who introduced it into his house in 1838. But Dr. Oesterling seems to have been the first one to see that this source of light and heat could be utilized in a large way. For a number of years he urged the formation of a stock company to drill for gas. But it was only after Pittsburg had demonstrated the value of this fuel to manufacturing industries that it was possible to carry out such a project. The first well was put down in November, 1884; the gas from it came with sufficient force to run an engine when turned into it. The Karg well was struck in January, 1886. It is claimed to have been the largest "gasser" in the Findlay district. Its estimated output was 12,000,000 cubic feet per day. The Trenton limestone gas resembles the oil from this source in containing odorous sulphur compounds.

32. Composition of Natural Gas.—The following is the analysis of Findlay gas as made by Prof. C. C. Howard :

Marsh gas	92.61%
Olefiant gas	.30%
Hydrogen	2.18%
Nitrogen	3.61%
Oxygen	.34%
Carbon dioxide	.26%
Carbon monoxide	.50%
Hydrogen sulphide	.20%

33. The large amount of gas that was found in Ohio led to the drilling of wells in Indiana. The gas found here, however, has been principally utilized for domestic purposes and the manufacture of plate glass. In Kansas, where new gas fields have recently been opened up, the gas is being utilized in smelting the zinc ores from Missouri. Among the other States in which gas has been found in large quantities might be mentioned Kentucky, Tennessee, Texas, and Michigan.

34. The waste of this valuable fuel within the borders of the United States can hardly be estimated. Jets of gas 10 feet across and 30 feet high were permitted to burn undisturbed for years, houses were heated to such an extent as to make it necessary to open doors and windows in midwinter in order to make them comfortable, and street lamps are still burning day and night in some towns.

THEORIES OF ORIGIN OF NATURAL GAS

35. Organic Theory.—The theory that petroleum is of organic origin is strongly supported by many American and European scientists, but the adherers of this theory do not agree as to the point whether petroleum is of animal or vegetable origin. Lesquereux and G. P. Wall are of the opinion that petroleum is due to the decomposition of marine plants, just as coal is the result of the decomposition of terrestrial vegetation. In contradiction to this, others claim that it is of animal origin and support their theories in many ways. Dr. Orton has made a very careful study of the subject, and his ideas, which are probably correct, may be summed up as follows:

1. Petroleum is derived from organic matter.

2. Petroleum of the Pennsylvania type is derived from the organic matter of bituminous shales and is probably of vegetable origin.

3. Petroleum of the Canadian type is derived from limestone and is probably of animal origin.

4. Petroleum has been produced at normal rock temperatures (in the American fields at least) and cannot be considered a product of the destructive distillation of bituminous shale.

36. Inorganic Theory.—Among the supporters of the inorganic theory are such eminent scientists as Berthelot, Mendeleeff, and Moisson. The latter materially extended the field of information in regard to carbon and its compounds and its reaction with metals, and advanced a theory based on the reaction of water with certain metal carbides. He says the reaction that appears to us most wonderful is the ease by which one can produce gaseous, liquid, or solid hydrocarbons by the action of cold water on certain metal carbides. The action of water on aluminum carbide gave pure methane and might be the cause of natural gas issuing from the earth in certain localities. A similar reaction may serve to explain the formation of liquid hydrocarbons or petroleum; in one case, 100 grams of liquid hydrocarbons was received from 4 kilograms of uranium carbide. To a great extent this mixture is composed of the ethane series, and only a very small percentage belongs to the acetylene series. At the same time, under ordinary pressure considable hydrogen and methane were obtained. This led to the belief that under pressure we would have nothing but methane or saturated series similar to petroleum. Doubtless during the early period of the world's existence the entire carbon was combined in the form of metal carbides. A sudden movement of the earth's crust would permit water to come in contact with these metal carbides and produce a sudden evolution of gas. The temperature would rise and produce polymerization of the hydrocarbons, and thus produce a very complicated mixture of these compounds. Through existence of metallic carbides, produced so readily by means of high temperatures, which are doubtless found in the earth's interior, one could readily account for the existence of gaseous, liquid, and solid hydrocarbons.

37. In giving a short review of these various theories and opinions, it is not the purpose to support one or the other, but simply to present them to the student as they now stand. A careful study of the subject, however, leads to the conclusion that the formations are, in all probability, not of a single origin, but that, for most instances, the views advanced by Dr. Orton are doubtless correct.

THE PRODUCTION OF CRUDE OIL

OIL WELLS

38. Location.—The drilling of wells is now systematically done by the most approved methods. The right to operate having been obtained (usually for a certain percentage of the gas or oil obtained), the well is sunk according to the opinion of the operator, although due consideration is paid to the wishes of the landowner.

In the early operations along Oil Creek all locations were made along the streams and ravines; as the hills were considered too high to produce oil, it was not until about 1870 that wells were drilled on the hillside as well as in the valleys. This soon developed certain defined lines known as *oil belts*, which have governed nearly all locations in Pennsylvania and West Virginia.

39. Construction of Rig.—The location being selected, the next thing in order is to put up a *rig*, or *derrick*. The first derricks were made about 34 feet high and were completely covered. The modern rig, as will be seen in Fig. 2, is of very simple construction, but possesses the necessary strength. Four strong uprights (2″ × 8″ beams spiked together) are held in position by cross-braces, and rest on heavy wooden sills. It is usually 82 feet high and 12 feet square at the base, the height depending on the depth to

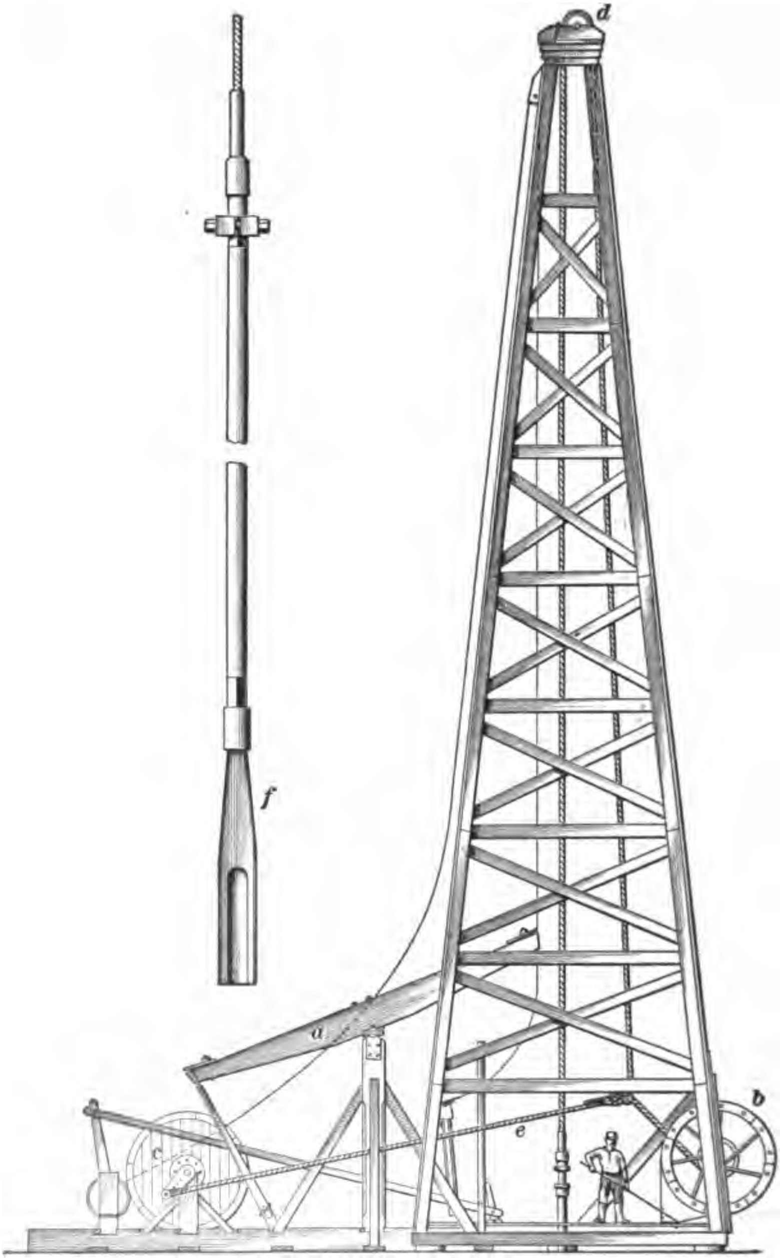

FIG. 2

which it will be necessary to drill. In shallow-well territory, a shorter rig will materially reduce the cost of operation. Connected with the rig is a long, shed-like structure, known as the *belt house* and *engine house*. This contains the engine and band wheel *c*, which communicates its motion through a pitman with the walking beam *a*.

It is customary for the owner of the well to furnish the necessary steam power; that is, a 25-horsepower boiler and a 12- to 15-horsepower engine, while the contractor furnishes the drilling tools, cable, blacksmith's outfit, and fuel for the boiler. Contracts are made at a certain price per foot, which varies according to the nature of the territory.

The old hollow sycamore that was used by the Ruffner Brothers in the first rock-bored well gave way to a smaller conductor, made of 2-inch planks nailed together to form a square 6 or 8 inches on the inside. A hole was usually dug to the bed rock, and this conductor placed in position as the rig was being built. This, in turn, has given way almost entirely to the drive pipe, which was first used by Drake in his well on Oil Creek. The first thing for the driller to do, after the engine has been set and all necessary connections have been made, is to drive an 8-inch or 10-inch iron pipe to the bed rock.

40. Drilling Tools.—This having been accomplished, the well is started by what is called the **spudding process.** It is usually carried out with light, short tools and a large bit, shown at *f*, Fig. 2, about the size of the internal diameter of the drive pipe. The tools are fastened to a short cable, which is passed over the crown pulley *d* and fastened to the *bull wheel b*. Now, by fastening a *jerk rope e*, which is attached to the crank of the band wheel, to one end of the cable, the tools may be raised and dropped at each revolution of the wheel. When sufficient depth has been reached by this operation to admit the use of a full string of tools, the spudding machinery is abandoned.

A good cable 2⅛ inches thick and 3,000 feet long (in deep-well territory) is passed over the crown pulley and coiled on the bull wheel. The end of the cable is secured in the rope socket, which is then attached to the full set of drilling tools, and these swung into the derrick. Their entire length occupies the space from the crown pulley to the derrick floor, and their weight is over a ton. They consist of a rope socket, 3 feet 6 inches long, weighing 80 pounds; 5½-inch jars, 7 feet 4 inches long, weighing 320 pounds; augur stem, 50 feet long, weighing 1,800 pounds; bit, 5 feet long, weighing 300 pounds.

The augur stem varies according to the nature of the country in which the well is being drilled. Formerly all drilling was done with a center drill, and the hole enlarged and irregularities smoothed up by means of a rimmer. Now, all drilling is done with a fluted drill, having a rather blunt point. By this change, the time of drilling has been reduced over one-half.

41. Everything having been carefully examined and adjusted, the regular drilling commences, and the machinery is kept in motion day and night. One driller and his helper, the tool dresser, work from noon until midnight, and another pair from midnight until noon.

The driller watches the jar, from which he can tell what his drill is doing. By means of the temper screw he lowers the tools and penetrates the rock; when the whole length of the screw has been run out, or the slow progress of the drill gives warning that it is working in hard rock and needs sharpening, he informs the tool dresser that he is ready to "draw out." The tools are withdrawn, a new bit put in place of the dull one, and the sand pump run down to remove the water and sediment that has been caused by the loosening of the earth and rock by the drill. This being accomplished, the bit is tightened with the wrenches and the tools lowered to continue as before.

While the driller is attending to his work, the tool dresser looks after the boiler, places the dull bit in the fire, and attaches a rope to the bellows handle; this rope is attached to the wristpin of the band wheel so that the fire is blown and the bit heated without further attention. Having oiled his machinery and attended to other odd duties, he returns to his fire and closely observes the "heat." As soon as the proper heat has been obtained he calls the driller, hands him a sledge, and together they hammer the end into the proper shape. The whole operation requires a thorough knowledge of blacksmithing, for the bit must not be overheated while in the fire and must be properly tempered after it is dressed. The entire work is such as to require strong, healthy, and experienced men.

42. Casing the Hole.—In order to prevent the water entering the oil strata, the well is cased as soon as the driller has reached the bottom of the water-bearing rocks. In some instances it is found necessary to put in several strings of casing on account of different veins of fresh and salt water. As soon as the casing is placed the drilling continues, but with a smaller sized bit—one that will enter the casing.

Before wells were cased, it was frequently almost impossible to tell whether or not an oil-bearing stratum was reached when drilling was suspended. If it was considered worth testing, a seed bag was put on to the end of the tubing and lowered into the well and permitted to stand several days. The water was then exhausted from the bag and the oil permitted to leave the rock. This method, however, was objectionable, for the seed bag was apt to burst and undo the whole work at the first sign of oil. Mud veins in the third sand on Oil Creek and at Pithole would stick the tools effectually before the wells were completed; but casing has proved an entire absence of anything like mud veins near the oil strata.

43. While the general principles concerned in the operation of drilling and in the construction of the tools have not been materially modified, labor-saving devices have been steadily introduced. Wells are now drilled in 15, 20, or 30 days, that in the beginning of the oil industry would have required as many months to complete. Great ingenuity is displayed by a professional well driller in contending with and successfully overcoming the innumerable obstacles, mishaps, and breakdowns with which he meets. The tools that have been invented on the spur of the occasion to meet some unexpected difficulty are to be numbered by the hundred. They are of all kinds—from the delicate grab designed to pick up a small piece of valve leather, or a broken sucker-rod rivet from the pump chamber, to the ponderous string of pole tools containing tons of iron, which, at a depth of 1,500 feet or more, can unscrew a set of stuck tools and bring them up piece by piece, or cut a thread upon the broken end of a sinker bar or an auger stem, so that it can be screwed fast to and loosened by the use of "whiskey jacks" at the surface.

44. Location of Strata.—In the early days of oil operations the dip of the rock was confusing to the driller. He could not understand why, if oil was found at a depth of 69 feet at Titusville, Pennsylvania, it could not be found at the same depth at Bradford, Oil City, and Butler. In fact, the territory around Bradford was condemned because oil could not be found at shallow depths. The Barnsdall well, which had been drilled to a depth of 200 feet in 1862, was deepened to 875 feet in 1866 without success. The driller once realizing that there were a number of sands and they were not to be found at the same depth throughout the country, proceeded to study this matter, and today is well informed regarding his particular work. His key rock in Western Pennsylvania is the limestone, which is from 5 to 25 feet thick in Butler, Armstrong, and Clarion Counties.

The following is an extract from a letter by a practical driller regarding his manner of estimation in the Butler County oil field:

"The limestone, which varies somewhat with the surface and location of the well, is used as our guide. The Butler County gas sand, commonly called **Berea grit,** is 875 feet below the surface of the limestone. This is the first oil-bearing sand of value in this section, with the exception of a few odd wells producing small quantities of pale oil from the 60-foot, which is from 50 to 65 feet from the limestone.

This is the sand that was formerly worked for brine. Following the Berea grit is what is known as the '100-foot,' which is about 1,000 feet below the surface." Table VI gives the different formations as they are known to the practical driller.

TABLE VI

Name of Sand	Distance Below Surface of Limestone. Feet
30-foot	1,150
Blue Monday, or Snee sand	1,200
Boulder	1,230
Third sand	1,250
Fourth sand	1,350
Fifth sand	1,425
Speechly	2,325

These depths, however, vary somewhat; in Washington County, for instance, the fourth sand was only reached at a depth of 2,640 feet, while in Armstrong County, the fifth sand was reached at about 2,000 feet, and the Speechly sand at about 3,000 feet. The latter is the productive oil sand of the Bradford field, where it was found at a depth of about 800 feet.

45. A drilling record of a well located in the Battelle District, Monongalia County, West Virginia, is shown in Art. **47.**

46. The Gordon sand of Washington County, Pennsylvania, and the West Virginia field is the fourth sand of the Butler-Venango field. This territory of deep and expensive wells has yielded some wonderful returns. But the expense has always retarded the developments. Only companies with extensive resources are able to hunt for new pools. A well in northern West Virginia costs about $9,500; in Washington County, Pennsylvania, about $8,000; in Butler County, $3,000; while on Oil Creek, many wells were put down for less than $500.

47. Torpedoing.—This method of adding to the productiveness of oil wells by the use of explosives is wholly American. The process was patented by Col. E. A. L. Roberts, who believed that the oil was contained in crevices not always reached by the bore hole. He proposed using nitroglycerine, or some other explosive, in the well to break up the rock and open these pockets. His idea met with disfavor at first, but he was finally permitted to make an

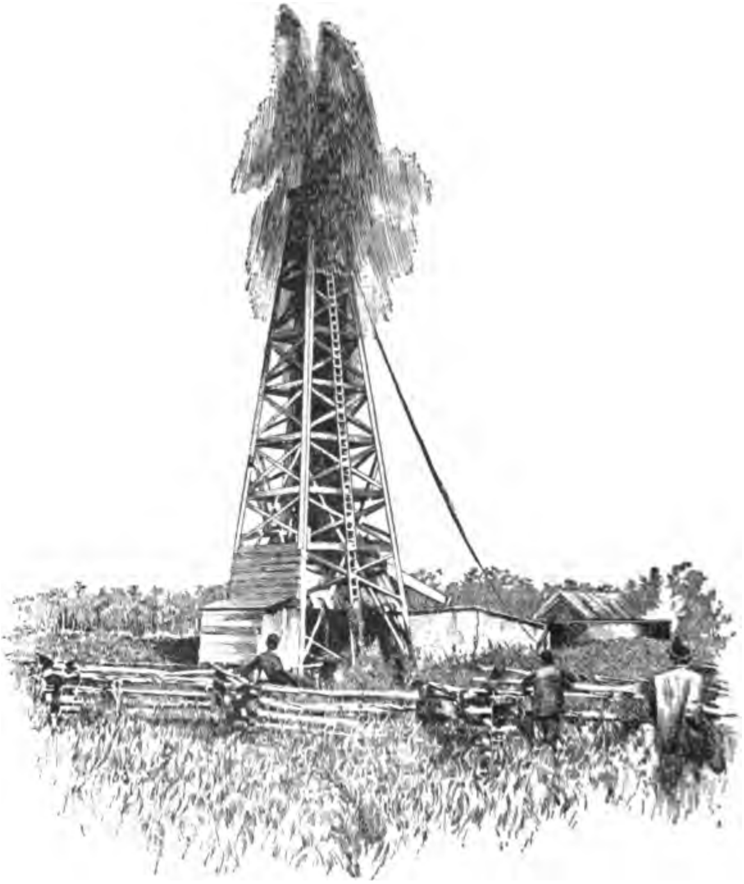

Fig. 8

attempt upon what was known as the "ladies' well," at Titusville, Pennsylvania, and obtained favorable results. He next tried it on the "Woodin" well, a dry hole on the Blood farm. His first torpedo secured a production of twenty barrels per day; within a month, a second torpedo was tried, which brought up the production to eighty barrels.

The most wonderful results ever produced by a torpedo, however, was that of the Armstrong well on Thorn Creek, Butler County. Fig. 3 represents an oil well shortly after shooting.

Veins Met	Distance of Top of Vein Below the Surface. Feet	Distance of Bottom of Vein Below the Surface. Feet
Pittsburg coal	1,306	
Dunkard sand	1,815	1,860
Gas sand	1,970	2,110
Salt sand	2,200	2,240
Indian sand	2,590	2,630
Squaw sand	2,800	2,920
Thirty-foot sand	3,080	3,100
Gantz sand	3,170	3,175
Fifty-foot sand	3,205	3,305
Gordon sand	3,512	3,538

THE TRANSPORTATION OF OIL

TRANSPORTATION OF CRUDE OIL

48. Oil Barrels.—The early method of transporting crude oil in America was in 42-gallon barrels, which were filled at the wells and hauled to the nearest railway station or wharf along the river. Oil Creek was used to such an extent that the amount of water in it was not adequate to carry the boats containing the oil. The shippers, however, resorted to a system known as *pond freshets.* A dam at the saw mill near Drake's well stored the water, which at the appointed time was permitted to escape, and carried the loaded boats to Oil City. Skilled pilots assumed the responsibility of delivering the oil to the larger boats at Oil City for conveyance to Pittsburg, at from $100 to $200 per trip. The teamsters and wagons were in such demand that a man with two stout horses could earn $20 per day. With continual use, the roads became a canal of mud from 3 to 4 feet deep. The hauling of a few hundred barrels not infrequently consumed so much time that the shipper, through the rapid fluctuations of the market, would sometimes not realize enough to pay the wagon freight.

The following is given by McLaurin as the expense per barrel of oil delivered at New York in January, 1866:

Government tax	$1.00
Barrel	3.25
Teaming from Pithole to Titusville	1.25
Freight from Titusville to New York	3.65
Coopering and platform expenses	1.00
Leakage	.25
	$10.40

49. Tank Cars.—Flowing wells soon taught the producer that the barrel system was not adequate to handle the crude in such cases. The Empire well alone produced more oil in one day than all the coopers of the nearby cities could produce barrels for in a week. The railroads laid switches and branch lines to the places that contained rich pools, and for transportation placed large wooden tanks, holding about 2,000 gallons, on flat cars. In 1871, these wooden tanks were replaced by cylindrical tanks made of boiler iron, similar to those in use today. Yet this still necessitated the hauling of oil several miles before the railroad was reached.

50. Pipe Lines.—In order to overcome this difficulty, pipe lines were proposed. The first successful one was laid between Pithole, Pennsylvania, and the Miller farm. This system was held in such disfavor, however, that it was only by a rigorous enforcement of law that the interference of teamsters, etc. was overcome. Other pipe lines were shortly laid to this point and loading racks *a*, Fig. 4, constructed, which enabled several men to load a train of tank cars in a few hours. The same system is in vogue today for short shipments and the transportation of refined oil.

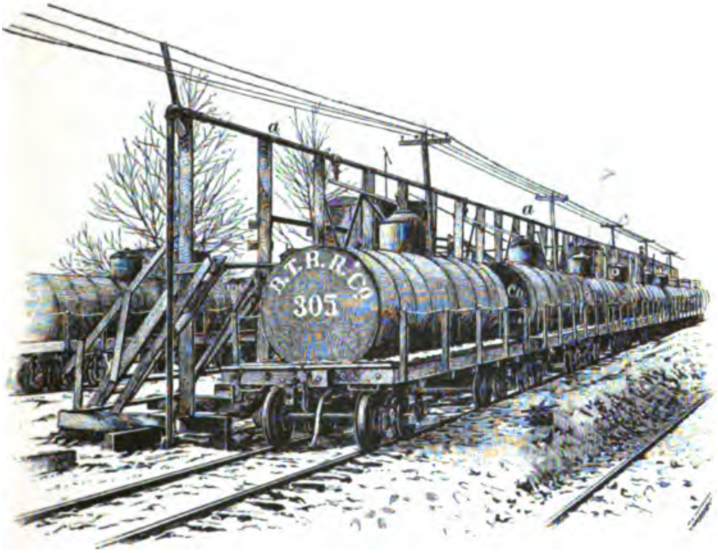

FIG. 4

Consolidation soon followed, and trunk lines were established. In 1875, a 4-inch pipe line about 60 miles in length was laid from the lower field (Butler County) to Pittsburg. The Bradford field needed especial attention on account of the large output and heavier crude. Improvements in pipes were made, and the first 6-inch line was laid from Tarrport to Carrollton in 1879. About the same time the first long-distance line was laid to Cleveland, and a trunk line to the eastern seaboard begun. At present, the oil field is a network of small lines, while several trunk lines traverse the whole length of New York and Pennsylvania, delivering thousands of barrels of oil daily to refineries, and loading tracks en route and at their termini at the seaboard. Refined oil, as well as crude oil, is now made in Cleveland, Oil City, and a number of inland towns, and transported through pipe lines to the seaboard, where it is loaded into immense tank steamers and sold in foreign markets.

51. The pipe-line system has been the means of saving millions of barrels of oil. When the drill approaches the sand, the pipe line is connected to the tank to take care of the production. In the case of some of the largest wells ever struck, the well owners were only provided with a 250-barrel tank, and it was only because the pipe-line company placed a large pump in position and forced the oil into the large mains, that the production was saved. Improvements in this system have been no less wonderful than in many of the other branches of the industry. The finished products are transported in tank cars to the large stations, from which they are usually sent out in barrels to the retailer.

TRANSPORTATION OF REFINED OIL

52. In order to accommodate the small jobber as well as large retail dealers, the refiners and railroads have compartment tank cars, as shown in Fig. 5 (this car has three compartments, each having a dome, as shown at *A*, *B*, and *C*). In such a car may be shipped kerosene, gasoline, and benzine, or different grades of kerosene. When oil is shipped in tank cars, the shipper is certain that the goods will arrive in the same condition as when shipped from the refinery. For short shipments of smaller quantities, everything is shipped in barrels. These barrels are usually of 50 gallons capacity and are made of the best oak staves.

53. **Preparation of Barrels.**—In order to render the barrels capable of holding the oil without leakage, they are coated internally with glue, about 1 pound of glue to four barrels being required. The hot-glue solution is poured into the barrels, the barrel bunged up with a temporary bung, and rotated so as to coat the entire inner surface; the surplus is afterwards drained out.

The glue having become hard, the barrel is painted with a cheap paint composed of a mixture of the cheapest color, benzine, and rosin. The rosin gives the barrel a hard, glazed surface and good appearance. Barrel painting is a process that requires considerable skill to do good work with the least loss of paint. A first-class painter is capable of painting a barrel per minute. In order to avoid the taint often caused by the glue of the barrel or leakage of the light gasolines, iron barrels are sometimes used. These, however, are only used for short shipments, where they can be returned promptly, since the barrel is too expensive for the receiver to own or for the refiners to lose.

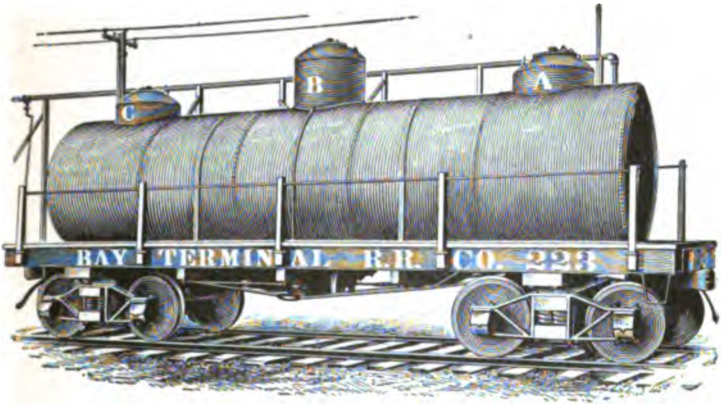

FIG. 5

At the present time all cities and larger towns are supplied in bulk and distributed by means of tank wagons, thus avoiding loss or contamination by barreling.

54. Shipment in Tin Cans.—Shipments of oil to warm climates or to distant parts are made exclusively in cans packed in cases. This avoids leakage and cheapens transportation. The cans are rectangular in shape, hold 5 gallons each, and are put up by twos in rectangular wooden cases.

PETROLEUM AND PRODUCTS

(PART 2)

REFINING PETROLEUM PRODUCTS

THE REFINING OF ILLUMINATING OILS

1. Introductory.—In spite of the fact that Samuel M. Kier produced a very poor illuminant when compared with the kerosene of today, yet he has the distinction of being the first to distil petroleum to be used for illuminating purposes. His "carbon oil," as it was called, sold for $1.50 per gallon; and by this means he disposed of all the petroleum the salt wells at Tarentum could produce. His operations, however, were of such a nature as to have but little influence on the industry when the crude oil appeared in abundance. The distillation of coal in retorts, and the manufacture of illuminating oils therefrom, which preceded the oil industry by but a very few years, was the real precursor in the development of the practical disposition of these enormous supplies of hydrocarbon oils.

2. Stills for Refining Petroleum.—In the refineries of the earlier days the stills were small, varying in capacity anywhere from 25 to 75 barrels. Many of these small stills are still in use, but stills constructed at the present time are much larger. The stills to be found in a refinery today belong to either of two models, viz., the *cheese-box* or the *boiler still.* The cheese-box still has a cylindrical body, flat

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steel-plate bottom, and dome-shaped top with outlet pipe at its highest point. They vary from 20 to 30 feet in diameter, and from 8 to 9 feet high. The use of this form of still is gradually diminishing on account of the longer time required in distillation and liability of accidents in the opening of seams in its bottom.

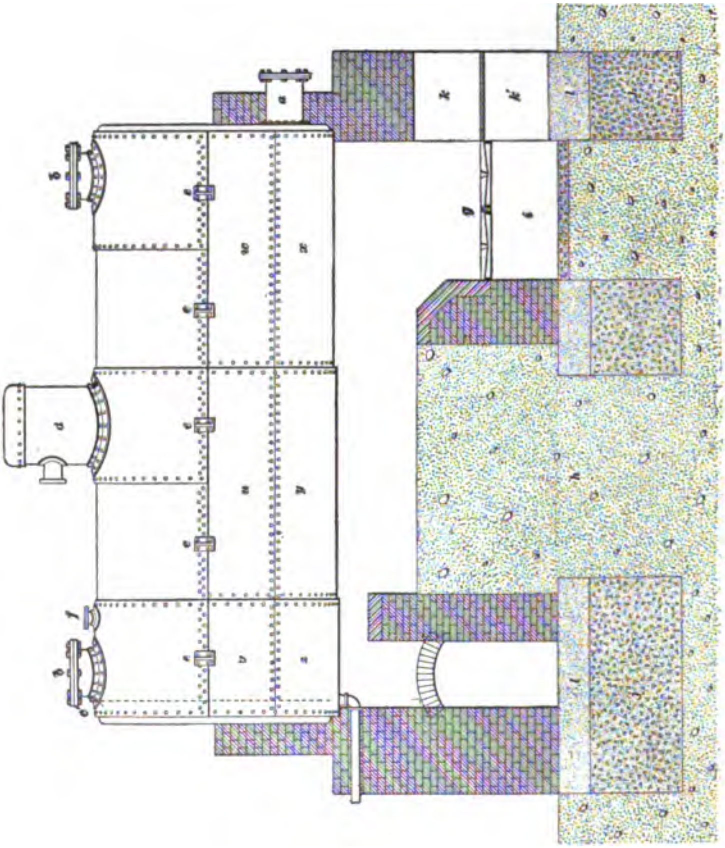

FIG. 1

3. Boiler Stills.—By far the most common still in use today is the horizontal cylindrical, or boiler type, shown in Fig. 1. The specifications for a 600-barrel boiler still are as follows:

Size of Stills: 12 feet in diameter and 30 feet long.

Dome: d to be 3½ feet high by 3½ feet in diameter and to have flat head.

Size of Material: Dome heads to be built of ½-inch flange steel, flanged. Dome shell to be built of ⅜-inch flange steel. Shell of still to be built of ⅜-inch flange steel. Bottom and side sheets to be built of ½-inch firebox or still-bottom steel. Heads of stills to be flat and built of ½-inch flange steel, flanged. Bottom of still to be constructed of three sheets; two, x and y, each 12¼ feet long and 8½ feet wide, and one, z (the rear sheet), 6½ feet long and 8½ feet wide. Side sheets w, u, and v to be 6½ feet wide and 6½ feet long. Shell to be made of five sheets, equal lengths, and of such width as to connect with the side sheets on each side of the still.

Rivets and Riveting: All rivets to be ¾-inch iron, Burden make. All bottom and side sheets and dome to be double staggered-riveted. Shell and heads to be single riveted.

Connections and Man Heads: The still is to be provided with one 18-inch wrought-iron neck man head a on the front end, having faced ring and plate and square-headed bolts and cover crane, the man head to be located in the center of head and about 15 inches above the bottom of the still; two 20-inch cast-iron man heads b with hinged bolts, faced plate, and top, each to be riveted on center line of top near each end; one 15-inch wrought-iron flange riveted to rear of side of dome, about 3 inches below the top of same, for condenser connections; one 6-inch wrought-iron flange f, for filling connections riveted on center line of top of still, about 6 inches forward from rear man head; one tar valve complete on bottom of still near the rear end, and having stem c' extending up through center line of top of still about 6 inches forwards from rear end of the same, having wrought-iron flange and stuffingbox.

Stayrods, Lugs, Etc: Each still is to be provided with 10 cast-iron brackets c, 5 on each side, same to be riveted on side of still, for the carrying of the still on the walls. Still to be provided with the necessary **T** bars and cross rods between heads to hold the heads firmly in place. This is to be accomplished by one **T** bar across the center of each head and by two $1\frac{1}{4}$-inch rods, with turnbuckles, running the full length of still. Also short **T** rods to be located above and below center **T** rods, having tie-rods extending from these and connecting with the sides of the still.

4. Stills are placed on good cement foundations *l*, Fig 1, and substantial brick walls; *g* are the grate bars; *k* is the furnace door, and *k'* is the door to the ash-pit *i*. The parts *h* are filled in with earth. The concrete *j* supports the cement foundation. About one-half of the still is exposed to the air (except for a sheet-iron jacket), to prevent burning of the vapor when it comes in contact with the walls of the still. The part exposed to the direct heat of the firebox should not exceed one-third the circumference of the still (12 feet, 6 inches). This is important, in order to protect the bottom of the still, since the level of the oil will come below that point at the end of the distillation and the iron will become overheated and warp. This will loosen the rivets and shorten the life of the still. Another important point is to guard against burning the vapor when the fire strikes above the oil line. In a well-constructed still all these points have been carefully considered. To the flange on the dome is attached a 10- or 15-inch vapor line. This vapor line leads to the condenser, at an angle of 65° to the surface of the still. Before entering the condenser it is customary to provide the line with a pressure and vacuum safety valve.

5. Condensers.—Condensers of many different types are in use; the most common form, shown in Fig. 2, is that in which the vapors traverse parallel pipes *a* of large condensing surface. The large 10-inch vapor line is divided into 4 4-inch pipes as it enters the condenser. The worm condenser is usually employed, but the old spiral form has given way to a worm composed of straight tubes connected

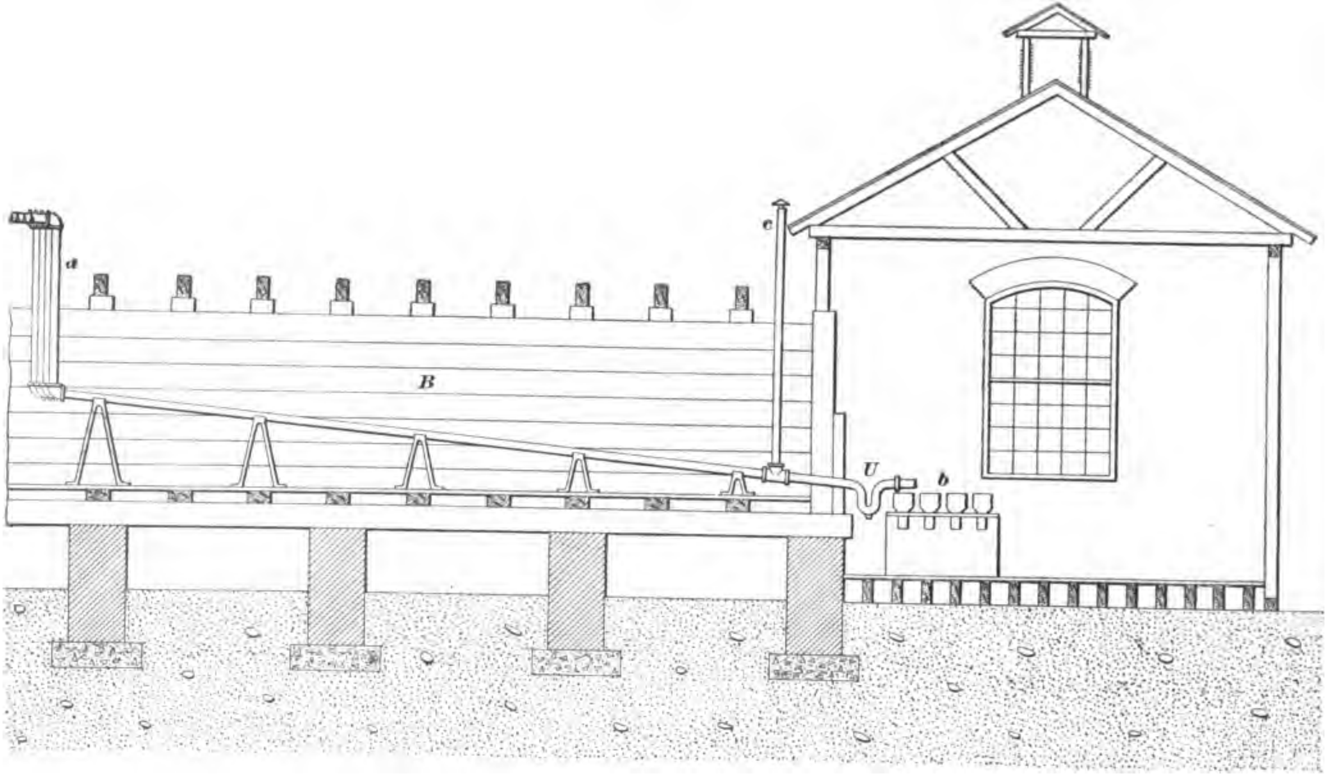

FIG. 2

by means of returns or elbows. These tubes or pipes are arranged in four parallel coils, with sufficient fall to prevent any condensed fluid from choking the passage within a rectangular tank *B*, made of iron or wood.

The common condensing medium is cold water. The water tanks are frequently large enough to accommodate a number of stills; the most convenient arrangement, however, where sufficient cold water can be obtained, is to provide each still with a separate condenser. The condensing surface required varies, of course, with the size of the stills, the grade of crude oil, and the products desired. For a 600-barrel still to be run for illuminating oil, where a large stream of cold water is at command, a condenser box, 40 feet long, 8 feet wide, and 6 feet high, containing a coil of 720 feet of 4-inch pipe and 576 feet of 3-inch pipe, has been found to be of the proper capacity. The supply of water should enter at the lowest point and the heated water leave the box by an overflow pipe.

6. The percentage of loss in the distillation depends in a great measure on the efficiency of the condenser. The water supply should be such as to admit the use of large quantities when necessary. It should be so regulated that even during the hottest summer weather all light distillates and illuminating oils do not pass the receiving house at a temperature exceeding 80° F. Formerly it was thought best to permit the water in the condenser to become heated while running off the illuminating oils. It was claimed that by this means distillates of a high fire test were obtained. The disadvantage of such an arrangement will be readily seen when we consider that the advantages are gained at the expense of a large loss of non-condensed light oils. At this period of sharp competition, refiners find that condensation must be as nearly perfect as it is possible to obtain in this way. Before the liquid leaves the condensing-pipe system to enter the receiving box, it passes into a **U** tube (see *U*, Fig. 2). The **U** tube forms a seal that prevents the passage of uncondensed gases.

In order to keep these gases from entering the tailing or receiving house, the pipe conveying the distillate has an upright pipe *c* attached before it reaches the building. The **U** tube prevents their passage, and the upright pipe discharges them from the line. These gases may be used as fuel, and in that case are drawn to the furnace by means of a steam jet at the point where they are to be used. This is of considerable economic value when operated properly, since fresh crude oil contains large percentages of gas dissolved in the oil, and these are always driven off with the first distillation; and again in the final distillation and coking process the heavy hydrocarbons are broken up and gases formed that cannot be condensed by ordinary means. These gases are an entire loss unless they can be used as fuel at the plant.

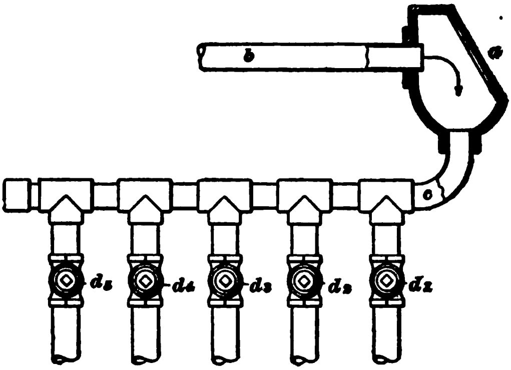

FIG. 3

7. After passing the **U** the oil enters the separating box *b*, which is usually provided with an automatic water tap that separates the water formed by the condensed steam and permits the oil to pass to the distillate tank. Fig. 3 shows the common separating box used in most refineries in the United States. It is a triangular-shaped, cast-iron box with a glass door *a*. The oil enters through *b* and passes out through *c*. Through the glass door the stillman observes the color and the size of the stream, and regulates his fires accordingly. From here, samples are taken and the gravity of the distillate calculated. The stop-cocks d_1–d_5 communicate with the receiving tanks that are usually under ground. The oil having reached the gravity at which it is to run to some other grade, the one stop-cock is closed and the other opened.

8. Receiving Tanks.—The Canadian method of constructing these underground tanks is to nail together in a cylindrical form pieces of 1-inch lumber about 18 inches long and 4 inches wide. These pieces are fitted together to almost fill the hole that has been dug. The earth being a blue clay is tamped solidly about this structure, and after being water-soaked from without and oil-soaked from within, makes a perfectly impervious tank. The tank is not affected by the temperatures of the seasons, and requires very little attention or repair, the only part affected by age being its roof.

9. Storage Tanks.—Most refineries are provided with large storage tanks for crude oil, in which it is permitted to settle in order to free it from water and other sediment. These tanks are sometimes located on an elevation, and permit the still to be charged by gravitation. More commonly, however, they are situated on a level with the works, or even under ground, and require large pumps to force the oil into the stills. To allow for expansion of the oil on being heated, the still is usually filled only three-fourths to four-fifths full of crude oil. The cylindrical still previously described is charged with 525 barrels of Pennsylvania crude oil.

10. Distillation of Crude Oil for Naphtha, Illuminating Oils, Etc.—The man heads and all lines connected with the still are carefully examined and closed. The fire in the furnace being kindled, is at first kept low in order to permit the fixed gases and moisture to pass off slowly. If moisture is present, it is recognized by a bumping in the still, becoming very severe if the firing is forced. With gentle firing, the oil is agitated without causing any injury, and the lightest gases and the water readily pass over through the worm. The quantity of water constantly increases until the water in the still boils. This is designated by the stillman as "the boiling of the still." The water soon decreases quite suddenly, and the stream at the receiving house slackens. The stillman then knows that all moisture has come off, when he says "the still has settled back." All this, however, will not take place when the crude is free from water.

The fires are now increased. As soon as the stream in the separating box has become clear and white, it is sampled and the gravity noted (in all this work the Baumé scale is used). The very first oil distillate, being colored and cloudy, is run into a separate tank to be rerun with a succeeding charge of crude. As soon as it has become clear and white and of proper gravity (75° to 95° Baumé), it is turned into the light naphtha tank by means of the stop-cock shown in d_1, Fig. 3. The stillman now observes the stream and from time to time determines the specific gravity. As soon as he arrives at the gravity at which he is to stop his light naphtha "cut," he closes this stop-cock and opens the one leading to the heavy naphtha or benzine tank. This cut is quite arbitrary, some refiners running all the naphthas into one tank. He continues as before and at the same time increases his fires slightly. Having arrived within a half degree of the gravity at which he is to begin the illuminating oil cut, he crowds his furnace to its full capacity.

11. In order to assist the distillation and produce a fine burning oil of sweet odor, the still is provided with a perforated steam pipe extending the full length of the still on the inside about 6 inches from the top. A very small quantity of steam is turned into the still, which helps to carry over the vapor more rapidly and prevents burning. When the stillman reaches the point at which the benzine is cut off, he directs the stream into the illuminating-oil tank.

This point is determined by local conditions, by laws regarding the burning point, and by commercial usage. The still is now kept running at full capacity until the stream reaches about 41° Baumé (this varies with the crude used), when it shows signs of color. The fires are slackened and the gravity will become lighter in the stream and the color will be preserved. This is the beginning of the so-called *cracking process.* The distillation is now continued into the high-test "water-white" distillate until a slight gas issue will be observed from the gas pipe outside the receiving house, which can usually be determined by several trial runs, and gauged by the stillman thereafter as so many hours after reaching 41° Baumé when the firing was reduced.

12. If the primary object of the refinery is the production of illuminating oils, one of two courses may be pursued: (1) The stream may be turned into a second-grade distillate tank and the oil run to tar of 19° to 21° Baumé. This mineral tar that remains in the still is permitted to cool to about 300° F. and transferred to a smaller still with extra-heavy bottoms and good internal bracings, to be again heated and distilled for paraffin and heavy paraffin oils. (2) It may be cooled after the first-grade oil is removed and transferred to a smaller still completely bricked in and having an elevated vapor outlet to carry out the cracking process, as previously explained. If, however, the crude oil is of such a nature as to permit the manufacture of good cylinder stock, a third course may be pursued after the first-grade illuminating oil has been removed, and that is the manufacture of wax oils and lubricating stocks.

13. As the average still attendant in a refinery is not a scientific man, it is necessary to give him exact directions as to the gravity at which to "cut" his oil in order to produce the grades required. This is sometimes quite difficult, on account of the great variety of requirements to which an American refinery is subjected. These cuts depend not alone on the crude oil, but more particularly on the market to which the salesman is catering.

These points are usually determined by making flash tests and fire tests of the different fractions and noting the quantity of distillate between each degree of gravity, in a run of the still to be used. After having arrived at a conclusion as to the grade of oil that it is advisable to produce, the still-man has simply to follow the directions as to firing and "cut" within the gravity limits prescribed.

14. Table I gives the results of a test made on a run of North Lima crude.

TABLE I

"Cut," Degrees Baumé	Distillate. Gallons	Flash Test
59–58	275	Flash below 59° F. in Tagliabue open cup
58–57	280	
57–56	275	
56–55	256	
55–54	300	60° F., Tagliabue open cup
54–53	285	73° F., Tagliabue open cup
53–52	290	75° F., Foster closed cup
52–51	315	93° F., Foster closed cup
51–50	375	95° F., Foster closed cup
50–49	360	109° F., Foster closed cup
49–48	400	112° F., Foster closed cup
48–47	310	122° F., Foster closed cup
47–46	335	135° F., Foster closed cup
46–45	298	144° F., Foster closed cup
45–44	306	158° F., Foster closed cup
44–43	280	163° F., Foster closed cup
43–42	295	172° F., Foster closed cup

Referring to Table I, the stream was allowed to reach 42° Baumé and the fires were permitted to burn lower in order to preserve the gravity and color. The distillation was continued in this way for 12 hours, the gravity fluctuating between 43° and 40° Baumé. All samples taken would flash below 60° F. in the open cup. This was caused by the cracking of the oil. All flashes showed signs and gave the odor of sulphur. The flash test of this oil was readily raised to over 200° F. by steaming or blowing hot air through it.

15. Spray Condenser.—Another class of condenser is what is known as the spray condenser. In this condenser the oil is mixed directly with a stream of cold water as soon as it leaves the main vapor line. This insures quick condensation and the use of the entire value of the cold water. The simplest form to be constructed is to use a 4,500-gallon cylindrical car tank and set it on end. The vapor line should enter the center of the head on top and extend down through about 18 inches into the water in the tank. Entering the vapor line as it enters the tank is a perforated water pipe, which is fed from a constant supply of cold water. The oil is carried to the tail house through a 3-inch pipe from near the surface of the liquid in the tank, which is about 18 inches from the top. The water is drawn off at the bottom, thus insuring a perfect separation.

This condenser is found to be especially valuable in the distillation of Ohio crude. Many believe that the water removes considerable sulphur from the oil, which seems entirely probable, especially where alkaline waters are used. The principal advantage, however, is the speed to be obtained by this method, it being almost impossible to burn any of the vapors where a good supply of cold water can be had.

16. Cracking Process.—The cracking process, as it is technically known, is the process whereby heavy oils and residuum are converted into specifically lighter oils, having a lower boiling point. This makes them suitable for illuminating purposes. The process is both of scientific interest and technical value. Peckham claims that there is only 10 per cent. of high-test first-quality illuminating oil to be found existing as such in the crude oil; but as much as 70 per cent. is made from the oil. Much of this, however, will be of a low fire test. The hydrocarbons standing between the kerosene and the lubricating oil, in gravity and boiling point, are of little value, and by this means may be converted into a salable article. Therefore, the proper manipulation of such a process is of great importance.

It is generally understood that the products of a fractional distillation, even in the laboratory, are by no means identical with the compounds existing in the crude petroleum. Professor Silliman, Jr., observed this fact in the irregularity of the boiling point in the distillates.

17. Thorpe and Young subjected solid paraffin to the action of heat under pressure, and thereby produced liquid hydrocarbons with but a slight loss as gas. A number of other chemists have conducted heavy petroleum through heated iron tubes and thereby produced lighter oils. The compounds produced have been found to belong to another series, viz., olefine, benzol, naphthalene, etc. This seems to convert the heavy American oil into the series most prominent in the Russian petroleum. The investigation of Professor Mabery in the distillation of California petroleum in a vacuum, which is just the reverse of the cracking process, has brought to light some new facts that may throw light on the compounds existing in the crude petroleum. As Mabery states, "none of the vacuum distillates nor the residue showed the slightest indication of decomposition; all portions had the natural odor of the constituents of the crude oil. From the lower fractions scarcely any paraffin could be separated."

18. In carrying out the cracking process, the principal object is to distil the oil at a higher boiling point than the normal. This object may be secured by having a large dome in which the vapor is partly condensed and made to fall back on the heated liquid; or, by elevating the vapor outlet, and instead of giving this line a fall, permit it to rise a very little before it reaches the condenser. A large number of arrangements for carrying out this process have been devised and patented, very few of which are in use today.

In most refineries little attention is paid to the matter, on account of the inferior quality of the oil produced by this means. Peckham lays stress on the following points, viz.: A pure, water-white oil of American origin is composed of the saturated-paraffin series, and on treating it with sulphuric acid the latter does not form compounds with the oil, but only with the parts which are foreign. Thus, after washing and neutralizing, nothing remains but the pure hydrocarbons.

In the "prime-white" oil, composed of the cracked oils, which is made up of olefines, the sulphuric acid finds compounds with which to combine, and on washing and neutralizing it will not be a pure hydrocarbon oil, some of the acid remaining in combination. Such an oil will readily darken in color, give off offensive fumes, and clog the wick, and cause endless trouble. In fact, here we have all the elements which produce complaint in the trade.

The only part of the process that is generally practiced is to reduce the fire and permit the exposed part of the still to carry out the partial condensation, and this falling back on the heated liquid may produce a closer fractionation if there is no decomposition. In most cases the oil remaining in the still is considered too valuable as a cylinder stock to be ruined in producing an inferior illuminating oil. In the distillation of the Ohio sulphur oils, however, this process has proved of great benefit.

The stocks remaining in the stills contain too much sulphur and other impurities to make a first-class lubricant without chemical treatment, and the breaking up of the heavy oils containing sulphur aids in their refining. Many patents have been taken out for the refining of Ohio oils, in which the salient point was really the breaking up of the sulphur compounds by a reheating of the vapor.

19. Distillation of Crude Oil for Lubricating Stocks. In a refinery aiming to make cylinder or other lubricating stocks from its crude oil there are some modifications necessary to the distillation described heretofore. The principles adopted are such as to avoid anything approaching the cracking process. In fact, every effort is put forth to preserve the heavy part of the crude oil in the same form as it exists in nature. In order to carry out this process, the distillation is assisted by superheated steam. This causes the oil to distil at a lower temperature and the admixture of steam prevents scorching from the heated still walls. The still as described in Art. **3** is provided with two 2-inch perforated pipes as shown in Fig. 4 at *a*, *a*, lying close to the bottom of the still. These pipes are about 1½ feet apart and reach throughout the entire length of the still. The perforations are made in two rows on opposite sides of the pipe with a $\frac{1}{16}$-inch drill. They are made so as to cause the holes to be turned downwards from a true horizontal position, thus causing the steam to strike the walls of the still and continually agitate the distilling liquid.

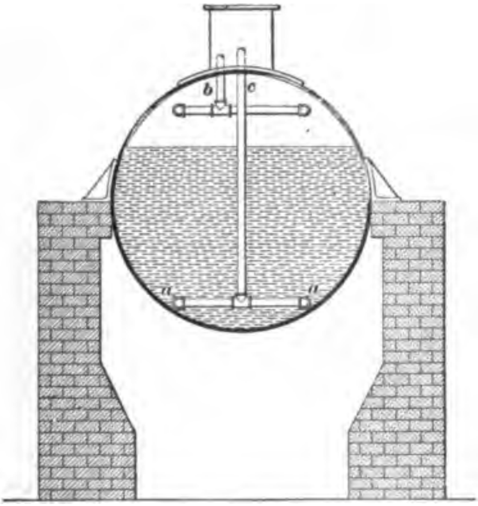

FIG. 4

Just outside the still on the pipe *c* is placed a first-class Fairbanks globe valve. This valve is certain in its action and does not permit any steam to pass when closed. The steam before reaching the still may be passed through a superheater, but if the still is close to the boiler house and a high-pressure steam is used, good results will be obtained without further superheating.

The still is charged and run as previously described with a little steam in the top line *b* until the point is reached where the first signs of gas begin to appear, showing decomposition of the heavy oil remaining in the still. Thus the crude has yielded its naphtha and first-grade burning oil in the usual manner.

In order to avoid even the slightest decomposition which will taint the lubricating stock, a trial run is made and the gravity of the oil in the still taken. All succeeding runs may be gauged by gravity instead of awaiting the signs of decomposition. In most grades of Pennsylvania crude oils, this point is close to 30° Baumé. When this point is reached, the fires are permitted to burn low. The moisture is all blown from the steam line entering the still through *c* by means of a small valve attached close to the valve on the outside of the still.

20. As soon as the steam has a blue appearance the valve on the line *c* is opened about $\frac{1}{16}$ turn. This will cause considerable commotion, and the stream at the receiving house will increase rapidly at once; but after a very few moments it will become quieted somewhat, when the valve is further opened to $\frac{1}{8}$ turn. The line conducting the top steam to the still is now shut off and the other valve opened wider from time to time until it is opened to its full capacity. If all lines leaving the receiving house have sufficient fall, the operation will require but a very short time. The firing may now be resumed and so regulated as to have a stream in the separating box, in which the oil and the water from the condensed steam stand about in equal proportion. If a superheated steam of 450° F. is at hand, it is well to keep the fires very low at the end. The fire test and viscosity of the oil depend entirely on the workings of the steam.

In order to distribute the steam throughout the still to better advantage, some have found it more satisfactory to have the bottom pipe constructed with a number of cross-arms, which are bent to the curve of the bottom of the still. By testing the oil in the still, it is seen that the fire test and viscosity rise as the gravity falls. The stillman again uses his hydrometer as his guide, and produces an oil of the required fire test and viscosity. When run with care, it seldom varies more than 2° F. in fire test from the same gravity of a previous run. The cylinder stocks used are either 600°, 650°, or 700° F. fire test. Some stocks for special filtering run as low as 500° F. fire test.

21. The distillate coming off after the introduction of the bottom steam soon becomes colored, and contains a large percentage of solid paraffin; the first part of it may be cut into a separate tank of 34° to 36° Baumé oil to be used in the compounding of miners' lamp oil, while the remainder, known as wax oil, is run into one tank and sent to the paraffin works. Having reached the required gravity in the still, the fires are drawn and the still pumped out as soon as advisable. If it is not pumped out very soon, the good accomplished may readily be undone. The vapors remaining in the still and settling back on the oil will give the latter a bad odor and taste, or if the steam is left in the oil after the fires are drawn, the moisture, which partially condenses on cooling, will make it unmarketable. Hence, to save the oil, it must be removed as soon as possible. In pumping out hot oil like this, the bottom steam is shut off and steam introduced in the upper line, so as to prevent any vacuum being formed in the still, on account of cooling and the removal of the oil. Destructive explosions have resulted from a neglect of this precaution, owing to the fact that the hot vapors of petroleum remaining in the still when mixed with air coming through the worm will ignite spontaneously.

This oil is marketed as steam-refined cylinder and grease stocks of various grades. When properly refined, all light oils removed, and no decomposition has taken place, the oil is perfectly tasteless and odorless. Much care is required in every part of a distillation of this kind. One of the most important features, however, is a constant and unchanging steam pressure and regular firing, so as to keep the still running an unvarying stream.

CHEMICAL TREATMENT OF THE DISTILLATE FOR BURNING OILS

22. Introductory.—The chemical treatment of the distillate derived from petroleum has an interesting history, the rank odor being one of the sources of trouble at the very beginning of its use. The first shipment of oil arrived in New York in 1857, and its strong odor was the cause of much complaint. The oil, besides having a strong odor, was of a yellow to dark-brown color and with age became darker. Its burning quality was very poor on account of the formation of a crust on the wick, which destroyed the capillary action of the fibers. In addition to these evils, the lamp emitted a very offensive smelling gas while burning. In spite of all these objections, oil was in such demand that the price rose to $2 per gallon.

In order to overcome these difficulties, a zinc-lined tank was constructed in which to chemically treat the oil. The tank was partially filled with oil and a hot solution of caustic soda added and the whole agitated with a wooden paddle. This was probably the first agitator used in America. The treatment was satisfactory in so far as it removed the smell, and the color also became lighter, but the oil soon turned dark again and deposited a solid crust upon the wick after burning a short time. It was next tried to free the oil from remaining lye by washing with dilute hydrochloric acid, but the color and burning quality remained the same.

23. Treatment With Sulphuric Acid and Caustic Lye.—Eichler, of Baku, is stated to have been the first to introduce, in Russia, the use of sulphuric acid and subsequent treatment with lye, and thus created a method which is in general use today. The action of sulphuric acid on the distillate has not yet been fully explained, but it appears to consist in the removal of aromatic hydrocarbons, petroleum acids, phenols, tarry products, etc. The manner in which these foreign products are removed is by no means the same in each case; some are partially destroyed or dissolved and changed to sulphonic-acid combinations, while others remain almost entirely unchanged and are removed only by the subsequent treatment with lye. The tarry matter in the distillate is dissolved by the sulphuric acid and imparts to it the dark color. The phenols, sulphonic acids, and the remaining sulphuric acid are neutralized and removed by the caustic soda. The acid is especially instrumental in removing the oxidized compounds of the distillate; in fact, by continuing the treatment all oxygen can be removed.

The action of the sulphuric acid is more complicated than appears at first sight. The evolution of sulphurous acid, which always accompanies a treatment, is not accounted for in the reactions given above. Its appearance can only be accounted for by the reduction of part of the sulphuric acid; but as to the compounds causing this reduction, nothing is known. That the acid actually attacks the compounds of the oil is true, since each succeeding acid will continue the action, evolve gas, and take on color. Its action does not seem to be limited. The longer the acid remains in contact with the distillate, the deeper will become the color of the latter. In fact, a certain color of the distillate in the treatment is one of the limits that the treater must not pass in order to produce the best results.

24. The quantity of acid required and the length of time during which the treatment is continued depend on the purity of the distillate, and this in turn depends on the composition of the crude oil and the manner in which the distillation was conducted. Cracked oils require more than the uncracked oils, and the Ohio sulphur oils more than the Pennsylvania oils. Usually the amount of acid required increases in proportion to the density of the distillate.

25. Agitator.—In order to secure prompt reaction between the oil and chemicals, they are intimately mixed by some mechanical appliance in a tank called the agitator. Those commonly used for the treatment of light oils consist of upright cylindrical vessels, with conical bottoms. They rest upon strong brick or stone piers, or upon an iron construction, as shown in Fig. 5. When located in a closed building, they are open at the top, but if erected in the open air, they are provided with a light iron hood, fitted with trap doors, to protect the oil from rain and from the direct rays of the sun, and still allow of a free circulation of the air.

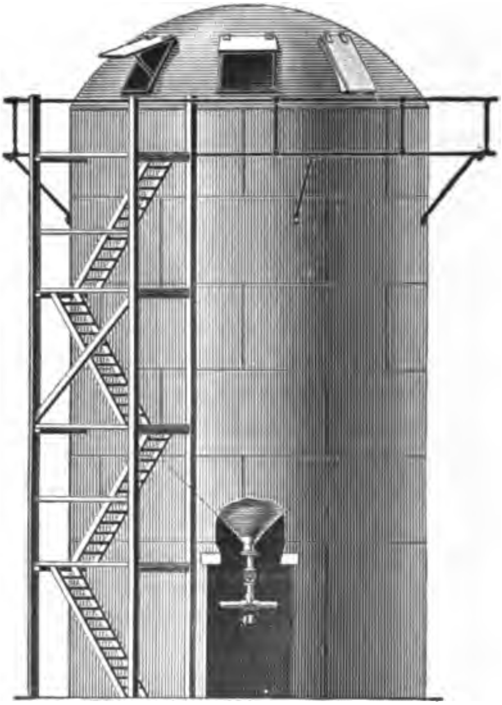

FIG. 5

They are constructed similar to all other tanks about a refinery, except that they have a lead lining to protect the iron from the action of the dilute acid formed when there is moisture in the oil, and also at the beginning of the wash with water. The thickness of the sheet lead is usually from $\frac{3}{16}$ to $\frac{1}{4}$ inch, but is sometimes as much as $\frac{5}{16}$ inch. The lead plates are laid on the sides of the agitator and lapped over the edges and soldered together, so that, as it were, they hang in the apparatus. It is customary to construct the agitators for light oils with a greater depth than width, because a more intimate mixture of oil and chemicals can be effected in a tall, narrow vessel than in a flat one. The cone is likewise constructed with steep walls, its depth being about two-fifths that of the diameter of the upper part. This allows a rapid settlement of chemicals, which is particularly important in the acid treatment.

Nearly all agitation is now carried out by means of compressed air, the air line entering the agitator at the top, and passing down to the lowest point of the cone. In addition to a pipe by which the agitator is charged with oil, it has separate pipes for acid, alkali, and water. The chemicals are forced in by means of compressed air. The bottom of the agitator is provided with a three-way stop-cock, with bores of large diameter to prevent their choking up by thick, tarry sludge, or it may be provided with any other suitable stopcocks and lines, as at *a*, Fig. 5, for discharging the acid, lye, water, and oil into their proper receptacles.

26. In making 150° water-white oil from Pennsylvania crude, the stillman uses the "cut" from about 58° to 40° Baumé. This will produce a distillate of light, lemon-yellow color and about 49° Baumé and about 110° to 115° F. fire test. In order to raise the fire test, the distillate is pumped to the steam still similar to the one used in rerunning naphtha. It is here steamed very carefully until all of the light oils have been removed and the distillate has the required flash point. It is now pumped to the agitator and cold water pumped through it until it has cooled to about 60° F. After standing some 5 or 6 hours, all the moisture will have settled out and is drawn off.

In oil of this grade it is customary to use about 6 pounds of sulphuric acid of 66° Baumé per barrel of oil. This is usually added in two portions, a little less than one-half being used the first time. This portion of the acid being drawn off into an air-tight drum and connected with the air compressor, the air blower is started and the oil put in agitation. The compressed air is then turned into the drum and the acid discharged into the oil. The agitation is now continued regularly and at such a speed as to keep everything intimately mixed. The attendent observes his oil very closely during the entire process. Samples are from time to time taken in a test bottle and the action of the acid judged by the manner in which it settles. In a very short time the acid is seen to settle in small, elongated drops, which adhere to the sides of the test bottle. Shortly after this the acid forms in small beads, which become larger as the process continues, and settle rapidly to the bottom of the flask. This is the point the treater desires to reach. The acid has accomplished its work and the blower is shut off. The operation thus far usually requires from 30 to 45 minutes. Should the temperature rise more than 2° or 3° F. before this point is reached, it is well to discontinue the operation, as a rise in temperature always indicates too violent action on the part of the sulphuric acid. This may be caused by moisture in the oil, or by too much moisture in the atmosphere.

27. After the acid has been permitted to settle for from 45 minutes to 1 hour, the sludge or spent acid is drawn off. The second or larger portion of the acid is now added in the same way as was the first and agitated about the same length of time. The treater will now observe that the oil assumes a cherry-red color and violet florescence, and that the acid settles somewhat slower; when, however, it has reached its limit, it quickly clears in the neck of the bottle, leaving the oil white. The acid treatment may be considered finished as soon as a sample of the distillate forms a milk-white emulsion when mixed with dilute lye; a series of these tests will soon show the practiced eye when the action is complete. Here, again, the same care must be taken as to a rise in temperature. It is well for the treater to wash a small sample to see what color it will produce.

The oil having been permitted to settle for at least 2 hours, the sludge acid is drawn off. The time of settlement depends very much on outward conditions and the distillate treated. The treater must keep a watchful eye on his oil during this time, and should he at any time observe any change in the color towards the violet, it will be well for him to wash a sample and note the results. The sludge should be drawn off every 15 minutes after the first half hour, for final results rest on the purity of the oil at this point. It is to be observed here that more complications arise from the use of too much acid than too little.

After all due precaution has been taken in the foregoing operations, the oil is washed with cold water. The water is pumped on to the surface of the oil in a fine spray and drawn off at the bottom. This is accomplished by means of a rose nozzle or perforated pipe running around the top of the agitator. It is well to wash down the sides of the agitator as soon as possible, for all traces of sludge must be removed quickly if a first-class oil is desired. This wash continues with intermittent agitation until all traces of acid have been removed and the water passes through the oil perfectly clear, and shows no signs of acid with litmus solution. The oil is now permitted to settle for some time (about 30 minutes) and the water drawn off. It is again agitated and 25 pounds of caustic soda, dissolved in water to about 6° Baumé strength, added in a small stream. This agitation is continued for about 20 minutes, when the oil should be thoroughly alkaline.

The oil is now permitted to settle for from 5 to 6 hours, when the alkali and soaps settle to the bottom and are drawn off. It is to be observed here that an undue amount of soaps and slow settling are signs of too much or too long acidification. The oil is now thoroughly washed with warm water of about 90° F. This wash is continued until the water flowing from the oil shows no signs of alkali on being tested with a red litmus solution. The oil is now transferred to large flat tanks, known as settling tanks or bleachers, where the remaining soaps and moisture are settled out. These tanks were formerly all so arranged as to enable the treater to permit the direct rays of the sun to act on the oil and bleach it.

28. The best illuminating oils are not bleached at present; they are usually settled by means of the steam wash. In this process, a perforated steam coil is placed in the bottom of the bleacher and the oil steamed until it reaches a temperature of 110° F., when it will settle out clear and bright within an hour. The oil is now ready for the market and may be shipped directly or placed in the storage tanks awaiting orders. Much has been done to produce marketable oils from the heavier and poorer stocks; but nothing has been found to equal a light cut of Pennsylvania distillate after a careful treatment as described.

In case of a treatment of cracked oils, which contain much more oxygen than the regular distillate, the acid process is not adequate to remove all foreign compounds with two applications. It is customary to use a preliminary treatment of concentrated lye of 25° to 30° Baumé. This removes the oxygen compounds, much tar and foreign matter, and gives the acid more opportunity for good action. Even when oils of good color and odor are produced from such distillates, they readily take up oxygen and deteriorate in color, odor, and burning quality.

29. Treatment of Oils Containing Sulphur.—Among the almost endless patented processes for removing sulphur from Ohio and Canadian oils, there is only one, the treatment with sodium plumbate, which has received the attention of all refiners. Although it has been discarded by many, and other methods used in its stead for a time, it is still more generally employed today than any other process. The only reason that refiners fail to make an absolutely acceptable oil by its use is the lack of intelligent and painstaking treaters. It is a delicate chemical reaction and should receive the attention of a perfectly trained eye and hand.

30. Sodium-Plumbate Treatment.—The oil is distilled and treated in the same manner as the Pennsylvania distillate just described, until the alkali treatment has been finished. Now, instead of washing the oil with hot water, it is heated with an open steam coil to about 110° F. and agitated with a hot solution (200° F.) of sodium plumbate, prepared as follows: Make up about 10 barrels of caustic-soda solution of 22° Baumé and dissolve in this 400 pounds of the best litharge. This is heated with steam almost to the boiling point. About 5 barrels of this is pumped into the hot oil and agitated for 20 to 30 minutes. The color changes to a dark brown, and at times to almost black. The treater tests his oil from time to time to see if it will stand the "doctor." To make this test, a sample of the oil is settled with a little flowers of sulphur and the clear oil decanted into a testing bottle. Now there is added the "doctor," a concentrated solution of sodium plumbate. The oil should not change color. If it turns a faint-yellow or dark-brown color, the lead-and-lye treatment is not complete. This state of affairs should take place quickly, and should it not stand the above test within 10 or 15 minutes after the beginning of this treatment, it is well to add more of the lead-and-lye solution immediately.

As soon as the oil stands the "doctor," shut off the air and permit it to settle. Within half an hour the greater part of the lead-and-lye solution can be drawn off and run back to the tank with the part remaining. As will be noticed above, we have prepared a large amount of chemicals, but most of these have been returned and can be used again. In the average treatment, about ¾ pound of litharge and ¾ pound of lye per barrel of oil are used. If the oil has been well treated, it will now settle out almost clear in the agitator. The more lye and moisture that can be settled out before proceeding, the better will the oil be when finished.

Some have washed it with water at this stage, which, however, necessitates a thorough settlement or reheating to remove the moisture.

The oil is permitted to cool to about 90° F. and the lead and lye drawn off as close as possible. It is again put in agitation and about $\frac{1}{12}$ pound of flowers of sulphur per barrel added in small portions. The oil is again sampled from time to time, and as soon as the black, flocculent particles of lead sulphide leave the oil clear in the neck of the bottle, it is finished. As flowers of sulphur is soluble in the oil, too much sulphur will destroy the good that has been accomplished in the lead-and-lye treatment.

The oil is now permitted to settle for 5 or 6 hours, when all the lead sulphide settles to the bottom, leaving the oil clear and sparkling. This sediment is composed of lead sulphide and a heavy tarry substance, doubtless compounds of the hydrocarbons, which have combined with the lead, lye, and sulphur. It forms a slight crust over its surface, which should not be disturbed again. It is necessary to draw off the oil by means of a swing line connected with the agitator above the cone, so as to leave all this sediment undisturbed. It is impossible to make an oil that will not clog the wick and leave heavy incrustations, if the black sediment is permitted to get up through the oil again after the first settlement. The sediment seems to act as a filter to the oil.

31. Grading of Burning Oils According to Color. The burning oils chiefly manufactured in the United States, both from Pennsylvania and Ohio crude, are usually designated by the fire test and color. The water-white oil of 150° F. fire test has been considered the standard oil in America for some time. Now it is necessary to call for a 48° Baumé gravity, 150° water-white oil, in order to obtain the high-grade oil, since we find on the market 45°, 46°, 47°, 48°, and 49° Baumé, 150° water-white oils. Besides the colorless water-white oils, there are all tests and gravities of *prime white*, a faint yellow; and *standard white*, a pronounced yellow. All these grades may be 110° oil of 110° F. fire test; 120° oil of 120° F. fire test; 150° oil of 150° F. fire test; and 175° oil of 175° F. fire test. Besides these brands, the gravity is looked upon as more prominent in the grading than formerly, and is receiving its just recognition in the production of a good, light oil.

The 175° oil should be of a water-white color and 45½° Baumé gravity. It is frequently sold under the brand of *headlight oil*, but all so-called headlight oil is by no means 175° oil. Its principal use is in the lamps of the headlights of locomotives. The United States Government frequently contracts for oil of 135° F. flash test, to be used as a safety illuminant for general purposes. Besides these grades, there is manufactured to a comparatively small extent a burning oil of 300° F. fire test, and of 36° to 38° Baumé gravity. Formerly, a standard white was sufficient, but at present it is required to be water white. This oil was first introduced by the Downer Kerosene Company, of Boston, under the name of *mineral sperm*. At present it is sold under the names mineral colza oil, mineral sperm oil, mineral seal oil, coach oil, and 300° oil. It is used for an illuminant in railway coaches, lighthouses, and for several other purposes where readily ignitible oils are objectionable.

THE REFINING OF NAPHTHA

32. Introductory.—If the crude oil is rich in the very lightest hydrocarbons, they are separated into various cuts as they come from the crude still. These crude naphthas, or benzines, as they are frequently called, can seldom be used as such, it being necessary to deodorize and refractionate them. Some of the lightest products are sometimes taken as they come from the crude still, such as 90°, 88°, and 80° Baumé gasoline. These products are used in the air gas machines for the manufacture of gas for lighting or heating, by passing air through gasoline. This mode of working in the refinery is not profitable to the refiner or the user of the gasoline, because the products thus obtained are less valuable, the carrying over of heavy products being unavoidable in the regular crude distillation. The best method is to make not more than two cuts at the crude still, viz., the light and the heavy naphtha, and refractionate these for high-grade gasolines.

33. Chemical Treatment of Naphtha.—The crude naphtha is pumped to the agitator to receive a chemical treatment similar to that which is applied to the oil distillates. For light naphtha, about 4 pounds of sulphuric acid of 66° Baumé per barrel is used, and all is added at once, unless there is danger of moisture, when it is well to use about one-fifth of the entire amount to remove the water, after which the agitation with the principal acid is continued for from 15 to 30 minutes, this time depending largely on the temperature of the atmosphere. If it is warm, the acid will take hold of the tarry particles very readily and the time is cut short in order to avoid loss by evaporation.

When the naphtha has been sufficiently agitated, the air blower is shut off and the heavier acid is permitted to settle for at least 1 hour, and even longer in cold weather. The sludge acid containing the particles of tar is drawn off after standing half an hour. After this it is drawn off about every 15 minutes, and when found to be free from sludge after standing 15 minutes, the naphtha is ready to receive the caustic treatment. Having dissolved about 15 pounds of pure caustic soda in water to a strength of 8° Baumé, the naphtha is again agitated and this solution forced into it. The agitation is continued for about 15 minutes and the mixture is then permitted to settle. After drawing off this caustic and dissolved soaps, a sample of naphtha taken from the top of the agitator and tested with hot water and a drop of litmus solution *must be free* from acid. The naphtha in this condition is called *deodorized naphtha*, since it has lost the penetrating odor which it had as a distillate.

It will be observed that the wash with water, after the acid treatment, has been omitted in the naphtha treatment.

The acid is so much heavier than the naphtha that it settles out very quickly and quite completely; the aim in the treatment is to shorten the process in order to save the naphtha, which is very volatile, to avoid a loss which may easily amount to several per cent.

34. Distillation of Naphtha.—The deodorized naphtha is now pumped, or, better, permitted to run by gravity, to the gasoline still, sometimes called the *steam still.* This is commonly a cylindrical still provided with a steam coil, similar to the bottom coil described under the cylinder stock distillation, only it is not placed on the bottom, but about 6 inches from it. This gives some space underneath the steam coil for the collection of water, which will not be disturbed during the distillation. The still is built similar to the other still described, with the exception that it is not necessary to make any distinction between the weight of the sheets used on the top or bottom, since fire is not used in this work. The still is placed on a good brick foundation and entirely covered over with a brick jacket.

35. Naphtha Still.—Many small stills have been devised that were capable of close fractionation, but in a gasoline works, where it is necessary to distil large quantities each day, a still constructed on the same principles as the one shown in Fig. 6 is used. The principal features of this still are a vapor line *a*, which has a high raise before it reaches the condenser, keeping back all particles of liquid that may have been carried along with the vapor, and what is known as a *back trap* for close fractionation.

The coil in the condenser *A* is attached at its lower end to a 6-inch pipe (the back trap), from which the line *c* (the back-trap line) leads to the bottom of the still. The line *b* connects with the condenser *B*.

As soon as the deodorized naphtha has been heated to the distilling point by means of the steam, the vapor gradually rises in the line *a* and enters the condenser *A*. It traverses the coil and a large fraction of the heavy gas will be condensed and return to the still by means of the back-trap line *c*; but the lighter gases, which are not so readily condensed, will rise and enter the condenser *B* through the line *b*, where they traverse a much longer condenser and become a liquid. Here the coldest water is absolutely necessary to prevent loss; and even then some gases will pass off through the line *d*. These may be caused to pass through a coil surrounded with a freezing mixture composed of ice and salt in order to condense the very lightest product, the *cymogene*. In this manner one is enabled to make a very close separation of light and heavy hydrocarbons. If 80°, 88°, or 90° Baumé gasoline is wanted, it may be separated here to a better advantage to both consumer and refiner than at the crude still.

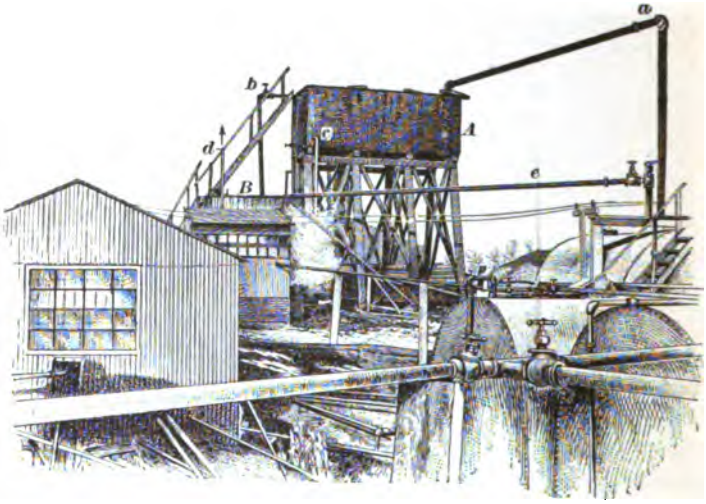

FIG. 6

36. In order to make the best products, the stillman must make his cuts by *weighing* (i. e., taking the Baumé density) both the tank and the stream. If the steam can be kept at a constant pressure, the still can be so arranged as to run practically automatically. Mr. Merrill, of the Downer Company, first obtained rhigolene, which has a density of 94° Baumé and a boiling point of 65° F. Cymogene may be obtained by vaporizing some of the very light products, as rhigolene, and condensing them by a freezing mixture. It has a density of 110° Baumé and boils at 32° F. These products are used in surgery as local anesthetics.

Some of the more common products manufactured are stove gasoline, from 68° to 74° Baumé, and a heavier product of 63° Baumé, known as deodorized benzine. Since the introduction of stoves whereby the former article can be used for cooking purposes, it has become one of the most important articles of the refinery. The deodorized benzine is used by paint manufacturers in the preparation of paints, enamels, and varnishes, and by pharmacists as benzine for cleaning purposes. This benzine should be free from oil and leave no sign of grease on a piece of white paper or silk.

Formerly much of the naphtha was fractionated and then treated; this has, however, given way entirely to the process given above. The redistillation after treatment leaves all chemicals behind and there is produced a pure hydrocarbon, perfectly safe to be used for any purpose for which it may be required.

37. The heavy naphtha, from 58° to 62° Baumé gravity, coming from the crude still, is frequently marketed without treatment as crude gas naphtha. It finds its use in such a case to enrich coal gas. If refined, it is treated in a similar manner to the other naphthas and rerun in the gasoline still. In rerunning this, the upper condenser is not used and the vapor is permitted to pass through the line *e* directly to the condenser *B*.

The whole naphtha is run into one grade, the 63° deodorized for paints, varnishes, etc. This still is used in the same way when raising the fire test of the distillates.

The naphtha from the sulphur oils is treated in a similar manner to those from the Pennsylvania crude, excepting the additional use of a small percentage of sodium plumbate. In the light naphtha this is found to settle out without the use of sulphur; but in the heavy naphtha it also requires some flowers of sulphur to clarify it similar to the oil treatment.

38. Tables II and III, taken from Redwood's "Petroleum," Vol. I, give the products manufactured by two different refineries from the light oils.

TABLE II

Product	Density. Degrees Baumé	Specific Gravity	Use of Product
Rhigolene or cymogene	90	.636	For surgical purposes as a local anesthetic.
Gasoline	88–86	.642–.648	For air gas machines.
Boulevard gas fluid.	76	.682	For street naphtha lamps.
Prime city naphtha (benzoline)	73–68	.692–.709	For sponge lamps, etc.
Benzine	62	.731	For oilcloth and varnish making.

TABLE III

Product		Density. Degrees Baumé	Specific Gravity
Petroleum ether.	1. Cymogene	108	.590
	2. Rhigolene	94–92	.625–.631
	3. Gasoline	90–80	.635–.668
Petroleum spirit.	4. C naphtha (benzine naphtha).	76–70	.682–.702
	5. B naphtha	66–65	.716–.720
	6. A naphtha (benzine)	59–58	.743–.747

39. All the products in the tables above sink into insignificance when compared with the stove gasoline, which in many cases consumes everything, from the very lightest to the heaviest, its demand having far outstripped the fondest hopes of many of our wide-awake refiners.

In speaking of the utilization of the light products of Russian petroleum, Crew says: "The light oils evaporated during the first distillation are allowed in most instances to run into the sea, but lately certain refiners have commenced to work them into gasoline and benzine, and, though the market for these products in Russia is limited, they yield a reasonable profit." This same thing can be said of the American light oils prior to about the year 1885. Then the naphtha was run into streams or used as fuel under the stills as a waste product.

Since it was first refined it has gradually worked its way to the favor of the public until, during the summer of 1898, all grades, from the very lightest to the heaviest, commanded a higher price than the best grade of kerosene. From being a by-product in the petroleum refinery, it has taken first place. Use has been found for it for cooking and heating, for whatever purpose gas could be used, and, consequently, it has brought comfort to people of the rural districts.

THE PARAFFIN INDUSTRY

40. Introductory.—Notwithstanding the fact that paraffin had been known for a long time, its manufacture on a large scale is of a comparatively recent date. It was known to exist as ozokerite, or earth wax, in Europe and various parts of America, but the discovery that it is a product of distillation of several organic bodies belongs to Karl Reichenbach, who gave it the name paraffin, from *parum* and *affinis*, because it appeared to be wholly destitute of chemical affinity. Its commercial value is based on the work of Seligue, of France, who manufactured it from bituminous shale. The production from this source has grown to enormous proportions in Europe and had taken a fair step in America when the more abundant and cheaper crude material, petroleum, took its place. In this country it is now entirely obtained as a by-product in the petroleum distillation, and this, like many of the other by-products of petroleum, has grown to such proportions as to become almost the principal product in certain petroleum distillations. It does not possess a constant composition; but, like all the other products of petroleum, it is a mixture of a number of hydrocarbon compounds.

In treating the subject from the standpoint of the refiner, the preparation of lubricating oils and the manufacture of paraffin wax run so closely together that in some points at least they touch, and must be treated as if they belonged to one subject. The manufacture of either or both begins with the distillation of the residuum coming from the illuminating oil distillation or the crystallization of the wax oils coming from the lubricating-stock distillation.

41. Distillation of Oil Residues.—The mineral tar or residuum is transferred after becoming entirely cold, or is reheated and then transferred into stills of about 300-barrel capacity. In the largest works much larger stills are also used. These stills have especially heavy steel bottoms and good internal bracing. The outlet, or vapor, line is large and is divided, by means of a **U**-shaped arrangement of the lines at the condenser, into two parts, as shown in Fig. 7. The one line *a* remains on a level with the vapor line, passes around the condenser, forming an air condenser of from 80 to 100 feet, and finally enters the condenser box and passes to the receiving house through a separate line;

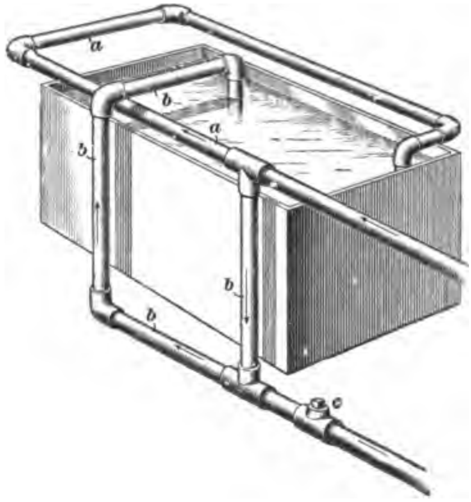

FIG. 7

the other line *b* makes a drop of about 5 feet from a **T** in the vapor line, and forming a **U**, rises again, enters the water box, traverses it several times, and thence passes to the receiving house. Thus, the still has two separate lines that carry off distillate at the same time. The water condenser is small and not kept too cool.

The still is heated gradually. If the tar has been chilled it begins by reporting with a bumping sound; this will increase in rapidity, but should not increase in force; if it does, it is a sign of moisture. A regular humming and crackling sound indicates boiling and no danger from moisture. This boiling may require 3 or 4 hours, according to the condition of the tar used. The fires are now crowded to their utmost in order to distil without cracking. The first distillates coming from both air and water condenser lines are run into a separate tank, to be rerun or used as a heavy gas oil. As soon as the oil coming from the water condenser line is clear and usually about 36° Baumé, it is turned into the paraffin distillate tank. The air line will run oil which is quite light and contains but little wax, and continues to run to the heavy gas oil, until near the end of the run, when it is found to contain wax and is turned into the same tank as the stream from *b*. Thus the stillman is enabled to separate the light oils from the heavy oils without refractionation.

The paraffin distillate is made in one long cut. The only point to be observed is to avoid too much color. The bottom of the still assumes a cherry-red color and the distillate becomes heavier and more waxy until the latter end of the run, when it is almost a pure wax. This is known as the *wax tailings*, and is permitted to run directly to a separate tank, by opening the stop-cock *c* on the vapor line. The whole charge of 240 barrels may be run off in from 8 to 12 hours. Since the still is run dry in order to get all oil and make a good coke, the stillman must be experienced in knowing just when to shut it down and not overtax the bottom, which with care should make from thirty to forty runs. The coke from the tar still is a valuable by-product, being used in the manufacture of electric supplies, artist's carbons, etc.

42. The other source of paraffin in a petroleum paraffin works, as already mentioned, is the wax distillate coming from the steam distillation in the manufacture of lubricating stocks. This wax oil is again placed in the still and rerun without the use of steam in the bottom of the still. This is what is known as the crystallization of the wax oil. Much has been said for and against the idea of paraffin existing in the crude oil in an amorphous state. However this may be, it is a known fact to the refiner that unless the oil be distilled at a high temperature the paraffin will have a vaseline consistency and stick to the disks and filter-press plates; whereas the crystallized wax oils press easily and the wax comes off in hard and firm cakes. The cut from the crystallization of the wax oil is the same as the first making of a distillate of 30° to 34° Baumé. This, however, varies, according to the peculiar views of the refiner and the wants of his customers.

43. Treatment of Paraffin and Wax Distillates.—The paraffin and wax distillates are pumped to the paraffin agitator (see lubricating agitator described later). This is constructed on the same general principles as the other agitators described with the exception of a special arrangement for heating its contents and keeping the dense and easily congealed oil in a liquid condition. No more heat should be employed than is actually necessary to keep the contents in a fluid state and the paraffin entirely melted. The same general principles of chemical treatment followed in the case of the illuminating oil are observed here. The action of the acid on the oil is very energetic and is accompanied by the disengagement of large volumes of vapors containing sulphur dioxide. For drawing off the heavy acid sludge coming from this treatment, exit pipes and stopcocks of large internal diameter are required. The sludge on standing becomes quite solid, and on being neutralized with lime forms an asphaltum which is excellent for roofing and light paving.

The acid treatment is followed by the usual water and alkali wash. This treatment being complete, the oil is transferred to a shallow tank provided with a steam coil, in order to keep the wax from congealing, and to permit the settlement of the water. This purifies the paraffin, removing all coke and petroleum acids, which are ruinous to the canvas of the filter presses.

44. Chilling.—The oil is next transferred to another shallow tank, where it is subjected to the chilling process. This was formerly carried out by means of a freezing mixture, composed of broken ice and salt placed about the cask containing the oil, similar to the arrangements in an ice-cream freezer. At present, ammonia ice machines are employed entirely for this purpose.

One of the methods in applying the cold produced by the evaporation of the liquid ammonia is to lead the ammonia pipes, together with a set of pipes to convey the oil to the filter press, through a vat containing a solution of calcium chloride. The evaporation of the liquid ammonia in the pipes, brought about by means of a vacuum pump, chills the solution of calcium chloride, which in turn cools the oil being pumped through the other coil, and thus causes the paraffin to crystallize. The wax oil may enter the vat at 80° to 90° F. and leave it at 10° or 15° F.

45. Pressing.—The first pressing is usually carried out at about 40° F. in a filter press (see Fig. 8). The chilled paraffin oil is placed in canvas bags and arranged in the filter press between perforated iron disks. The whole is subjected to a gradually increasing pressure, which forces out the liquid oil and leaves the solid paraffin behind. Unless this operation be skilfully conducted, enough crystallized paraffin may be forced through the canvas to interfere materially with the cold test of the oil.

These cakes of paraffin are melted and the fluid paraffin filtered through bone-black filters to remove the coloring matter contained in it. It is again chilled and placed in the hydraulic filter press, which is similar to the first, in which it is subjected to a much greater pressure at a higher temperature than before. It is pressed for from 5 to 8 hours at 70° F. and at a pressure of 300 pounds to the square inch. This produces a wax melting at from 117° to about 120° F. The melting point varies somewhat with the gravity of the oil brought to the presses. The cakes thus formed are removed and ground up into small scales. This scale when not treated with bone black is what is known as *crude scale*, and much of it is sold in this form to be worked up for special purposes.

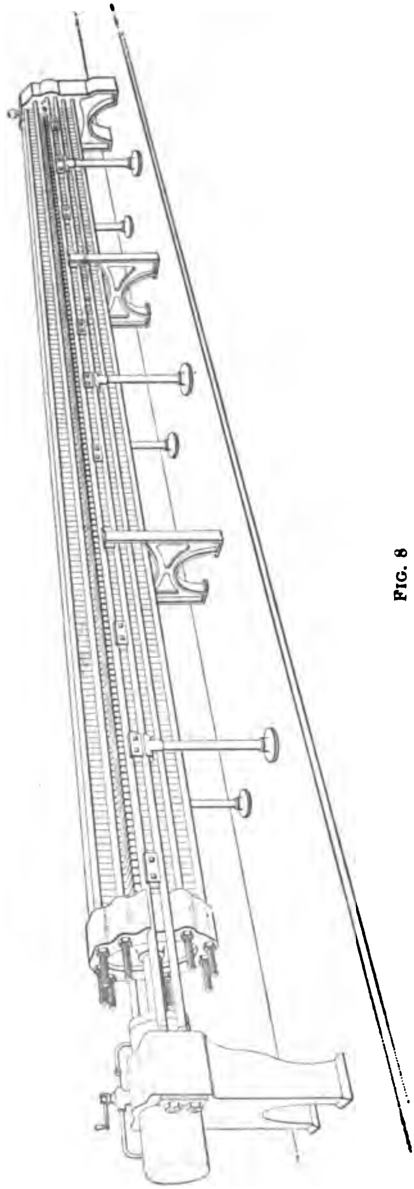

FIG. 8

The refiner making refined wax grinds the crude scale with benzine and by gently heating dissolves the wax. This is again cooled and subjected to hydraulic pressure. This is known as the *recrystallizing process*. The paraffin thus obtained is in large crystals, perfectly white and transparent, and has a somewhat higher melting point. Where there are no particular melting-point requirements this wax is used exclusively. In order to meet the requirements of higher melting points and perfect freedom from oil, the paraffin is put through the *sweating process.*

46. Sweating Process. — The recrystallized wax is melted and run into shallow pans 3 inches deep, 4 to 5 inches wide, and about 24 inches long. It is chilled and these cakes placed in a small room surrounded by steam coils. The cakes are placed on racks having perforated bottoms, underneath which are tin troughs leading to a common receptacle. This room is heated to the temperature of the melting point that the wax is required to have and kept at that temperature for about 12 hours, when all the oil and low-melting paraffin will have dripped out of the cakes, which come out perfectly honeycombed, but of the desired melting point. The American paraffin is usually sold at the melting points of 125°, 128°, and 135° F., known as C, B, and A paraffin, respectively.

Since paraffin has been produced in such large quantities from the American petroleum, the large manufacturers have made especial efforts to introduce it into general use. By their persistent efforts in informing the public and placing it on the market in convenient forms, it is steadily but surely becoming a household article of inestimable value. The paraffin candle has completely superseded all other forms of illumination along this line. It is also taking the place of sealing wax in the preservation of jellies and fruits.

THE PRODUCTION AND MANUFACTURE OF LUBRICATING OILS

47. Introductory. — The pecuniary and beneficial advantages accruing to modern civilization from the introduction of the heavy products of petroleum for the lubrication of machinery are hardly second to those resulting from the introduction of the cheap and beautiful light furnished by its illuminating portion. It seems strange that the second oil well in Pennsylvania should produce a petroleum so different from that found at the Drake well as to turn the attention of the promoters of the new field towards an entirely different industry. Such was the case: light and lubrication presented to the world simultaneously, one on Oil Creek, the other on French Creek, and both in a region comparatively isolated.

James Evans, a blacksmith at Franklin, drilled a hole in his water well, which had shown signs of oil at each recurring dry season, and at 72 feet struck a crevice. By means of a hand pump he succeeded in pumping up 25 barrels a day; and afterwards with steam power 70 barrels per day. It was a heavy oil, about 30° Baumé, free from grit and quite smooth. For some time the heavy oil was used principally in its natural state. By degrees, improvements were introduced and articles of a very high grade were produced by refining.

Joseph C. Sibley was the first to conceive the idea of manufacturing an oil from petroleum, to be used in steam cylinders. To-day engineers are enabled to carry higher steam pressure and operate with much less wear on the steam valves through its use.

Since the time that the Franklin oil was recognized to be of particular value as a lubricating oil, producers have been keen to examine carefully all heavy oils that might be found in any of their wells. Few, however, have found anything equal to that at Franklin. A field of considerable importance has been that at Mecca, Ohio. The wells are rarely over 100 feet deep; the oil has a density of 26° Baumé. Smith's Ferry, in Beaver County, Pennsylvania, has produced considerable oil of a peculiar amber color and about 28° Baumé. A number of shallow fields of West Virginia have been quite productive of heavy oils of excellent quality. One of the most noted was that at White Oak, West Virginia, where an excellent oil of 25° Baumé was produced at a depth of 160 feet. Nearly all the West Virginia oils are marketed without refining.

48. All heavy oils are by no means natural lubricants. Before being able to pass judgment on an oil, one must determine its purity, cold test, and fire test. The purity is estimated by mixing an average sample of the oil with an equal quantity of benzine, heating to about 120 F., and letting it settle for 6 hours in a graduated vessel. This gives a separation of all water and earthy matter. In Indiana, a heavy crude oil of 12° to 15° Baumé was produced containing large quantities of asphaltum, which rendered it practically valueless as a lubricant.

The variation in the cold test is quite marked in the heavy oils. The lower the cold test, the more valuable the oil. In this respect the oils from Mecca and Franklin are superior to all others.

A natural lubricating oil to be of real value must possess a fire test of at least 325° F. This is quite important, and many even of the low-gravity oils will not stand the requisite fire test for their employment in places where the inflammable vapors induced by friction would be dangerous. It has, therefore, been found necessary to bring these oils up to the standard by means of the processes called *sunning* and *reducing*.

49. Sunning.—In this process, heavy crude oils of 30° Baumé and under are selected. For this treatment, large, oblong tanks of about 18 inches in depth are employed. These are partially filled with water (about 6 inches) and the crude oil run in on this and exposed to the action of the sun and air for some time. This reduces the density and raises the fire test. It is one of the simplest processes to which natural oils are subjected.

50. Reducing.—The reducing process is similar to the foregoing. The large tanks are provided with steam coils, and not more than 4 or 5 inches of oil is placed on the water. The oil is heated to 110° F., when it becomes very limpid and every particle of impurity quickly subsides. The light oils evaporate and a crude of 32° Baumé is reduced to 29° Baumé in 2 or 3 days. The loss by evaporation depends on the gravity and nature of the oil; if the crude contains much light oil, such processes will be too expensive, and therefore impracticable. In such cases it will be found better to place the oil in a still and carry out the process described under "Lubricating Still." Nearly all heavy cylinder oils are produced in this way.

It is found quite practicable to reduce oils of 30° and 32° Baumé in a still, since their value is enhanced to such an extent as to command an increase of several cents per gallon over the crude article. The proportion of natural lubricants, in comparison to those that have passed through a refining process, is very small. Under the latter head there are the steam-refined stocks, already described, and the treated and reduced distillates.

As previously mentioned, the manufacture of distilled lubricating oils and paraffin is really one industry. The wax oils and paraffin oils after pressing constitute the basis of all grades of treated machine oils. The wax oils are usually cut into approximately the oil that is wanted, when they are rerun to crystallize the paraffin. The common cuts are 38° to 34° Baumé, and 33° to 30° Baumé. The first cut is sometimes used without being pressed, as a filler in the manufacture of soap. The requirements for such an oil are freedom from acid and lye, 300° F. flash, and a pale straw color. When pressed, the oil is used as a basis for compounding miners' lamp oil. The State requirement for miners' oils is one of gravity; the requirement for this oil is a good color, cold test, and free-burning oil, and not lighter than 34° Baumé.

51. Neutral Oil.—The second cut of 33° Baumé is pressed and then reduced in a still, by means of fuel and steam, to about 32° Baumé and 400° F. fire test. The oil is again treated with acid and alkali in the lubricating agitator and filtered through bone-black filters. The finished product is known as *32° neutral oil.*

The first neutral oil was made by Merrill, of the Downer Oil Company. It received its name from being as nearly neutral as it is possible to make a petroleum product. Merrill mixed 95 per cent. of his neutral oil with 5 per cent. of sperm oil, and thereby produced an oil in which the mixture could not be detected in color, odor, or taste. Oils of this type are used as mixing oils and for the lubrication of light machinery. Since the florescence of mineral oils is quite pronounced, it is common to expose these oils for some days in shallow tanks, in order to avoid their detection when used as mixing oils with vegetable or animal oils.

52. Paraffin Lubricating Oils.—The oil from the tar stills being run into one tank and pressed, is again rerun and cut into several fractions as paraffin lubricating oils. The common cuts are an oil of 29° to 30° Baumé, and a heavy cut of 23° to 26° Baumé. In order to give the oils a better fire test and viscosity, they are cut from ½° to 1° lighter than wanted and then reduced in the reducing still by means of steam and fuel. Thus the first cut is reduced to 28° Baumé of about 400° F. fire test, and the second made into 26°, 24°, and 23° Baumé oils, and 400° to 460° F. fire test. These paraffin oils are now pumped to a lubricating agitator.

53. Lubricating Agitator.—This agitator is similar to the one used for the treatment of illuminating oils, but is further provided with a steam coil for heating, and is usually surrounded with a brick jacket. Instead of carrying out the entire treatment in one agitator, it is customary to use two, one for the acid treatment and the other for the lye treatment. The cone is quite shallow and has large draw-off pipes to remove the acid sludge. The oil coming from the reducing stills is permitted to settle for some time to free it from sediment and moisture. The amount of acid used depends on the color desired, and the viscosity and gravity of the distillate; it varies from 4 to 15 per cent. of the oil treated. The average temperature for the treatment is 70° F., and it should not rise above 90° F.

The same rules hold good here that were to be observed in the illuminating-oil treatment; according as a high or low temperature is used during treatment, so will the resulting oil be either dark or light in color. The method of treatment varies according to the preference and experience of the treater, some using all the acid in one charge and others dividing it into several portions. The following is a short description of a treatment in which the acid is divided into two portions.

54. Acid Treatment.—The oil is permitted to settle until it becomes transparent, and the water and sediment are drawn off; it is agitated and about one-third of the acid to be used added in the usual manner. The agitation is continued for about 1 hour. The sludge will soon gather in lumps, and after settling for 2 or 3 hours is drawn off as close as possible. It is wise to draw off this sludge as soon as possible, as it becomes absolutely solid after a short time.

The second acid is added in the same manner as the first and blown for about 1 hour. Some permit a settling of from 4 to 5 hours after the second acid has been blown, while others add 10 to 20 gallons of water to the 100 barrels of oil and thus throw the sludge acid out while the oil is still in agitation. The latter process is certainly much quicker; in either case the oil must be pumped from the acid agitator to the lye agitator. The sludge adheres to the sides of the agitator in such a manner as to make a good separation impracticable. A good wash cannot be carried out without great loss, and the addition of lye with the acid still remaining will bring the tarry matter into solution again.

55. Alkali Treatment.—The alkali agitator is constructed similarly to the acid agitator, and where practicable is placed at a lower level than the latter, in order to permit the oil to run from the former into it.

The neutralizing process with caustic soda is one of the most difficult processes connected with the treatment of lubricating oils, in which experience alone is a sure guide. The oil is agitated and 3 to 4 per cent. of caustic soda of 3° to 5° Baumé is added.

The oil loses its violet color and becomes a golden yellow as soon as it is neutralized. Samples taken out from time to time show signs of settling, and as soon as a good separation takes place readily, the agitation must be stopped.

The soaps are permitted to settle and, unless an emulsion has begun to form, can be drawn off in about 10 hours. During the agitation, one should test frequently for acidity and alkalinity. It is well to have the oil slightly alkaline, but still it must be remembered that a perfect neutrality is the point finally desired. When the settled soaps have been drawn off the oil is washed with hot water of 150° F. The aim is to leave it perfectly pure, and the washing must be continued until the water comes off perfectly clear and neutral. Great care and judgment must be exercised on the part of the treater to prevent loss. The oil is now run into large, flat settling tanks heated with steam coils, where it is kept until every particle of water has subsided and the oil has become perfectly clear and transparent. Agitation with warm, dry air assists greatly in this.

For light-colored and pale oils it is necessary to bleach them in shallow tanks or filter them through bone-black filters. The bleaching, or *deblooming*, as it is technically called, is carried out by similar methods to those already described under the heading "Chemical Treatment of the Distillate for Burning Oils." By this exposure its gravity is increased and its fire test lowered. Many so-called debloomed oils have not been subjected to this treatment, however, but have been debloomed by the addition of nitronaphthalene; such oils are severely condemned by engineers, as being ruinous to machinery. In order to still further improve the color of the oil, it may be passed through a bone-black filter in the filter house.

56. Filtering by Use of Animal Charcoal.—One of the modern appliances used in the production of high-grade lubricating oils is the animal charcoal filter. The filter house usually contains a number of filters arranged in benches, some of which are heated with steam and others by means of natural gas or hot air. Tanks to be heated with steam are usually double-walled with a steam chamber between, while those to be heated with gas jets are suspended in a brick wall with a hollow space about the tank. In the latter case the entire space about the tank is heated, instead of applying heat directly to the tank. This avoids any overheating of the sides of the tank, which would injure the oil. By means of the hot-air system, flue gases may be utilized to good advantage.

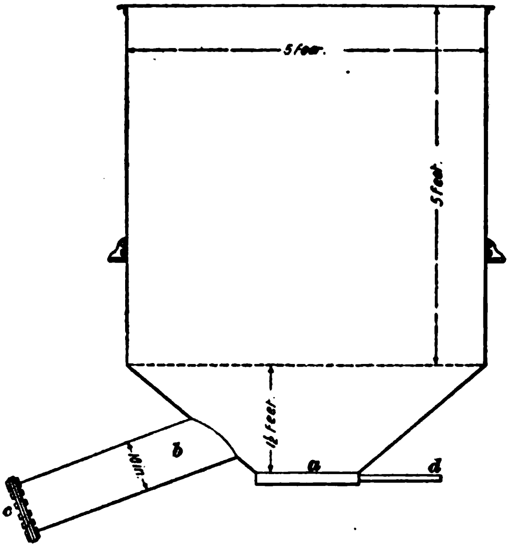

FIG. 9

57. Filters.—The tanks or filters are of two types, the side dump and bottom dump. Fig. 9 represents a side-dump tank. This is a well-riveted iron tank composed of $\frac{3}{16}$-inch iron. It is 5 feet in diameter and has a cone-shaped bottom with a perforated plate *a* about 4 inches above the outlet leading to the receiving tank. On the side, as represented, is a large pipe *b* reaching out from the tank, with a plate *c* bolted on to a smooth-fitting surface. By removing this plate the contents of the tank may be readily taken out.

The perforated bottom is covered with a thin layer of cotton batting and over this is about 2 feet of bone black. The oil is run in a slow stream on to the bone black, through which it filters and is then carried off through the 1-inch pipe *d*. It is provided with an automatic filler to prevent overflow in case the outlet of the filter should not act as fast as the feed. The oil is kept at about 120° F., which keeps the heavy oils perfectly limpid and aids the action of the bone black on the coloring matter contained in the oil.

The first oils coming off are always of a very light color and unless wanted of this shade for some special purpose it is mixed with the darker oils following, to some uniform color desired. As soon as the bone black ceases to accomplish the decolorizing wanted, a small car is run in alongside the filter, the plate on the side dump removed, and the bone black permitted to flow into the car, and the filter charged with a fresh lot.

58. Wash Filter.—The bone which has been removed is taken to the wash filter shown in Fig. 10. This, as is shown, is similar to the other filter except that it is stronger and provided with a screw-top man head *d*. The bone black is put into this and benzine forced in on it through the pipe *c* and permitted to filter through it and pass off to a tank or steam still through the 2-inch pipe *b*. This washing is continued until the benzine comes off perfectly water white. Steam is now forced into it through the pipe *a* and all benzine distilled off. The bone is now removed and put into cast-iron retorts and heated to dry and remove other impurities. This heating is very intense, but the admission of air is avoided.

This latter operation is so arranged that bone black is fed into the retorts and removed as a continuous process. In this manner the bone black is renewed, after which it acts as well as when used for the first time. Good bone black has thus been renewed as often as 116 times before it was found to be useless. When it is found to be impracticable to attempt a further renewal of the bone black, it is sold to fertilizer factories. The benzine which has been used for the wash is distilled with steam and the residue used as a cold-test black oil. It is possible to obtain any shade of oil that is desired, from the perfectly white neutral oils to the finest grade of colorless vaselines and petroleum jellies.

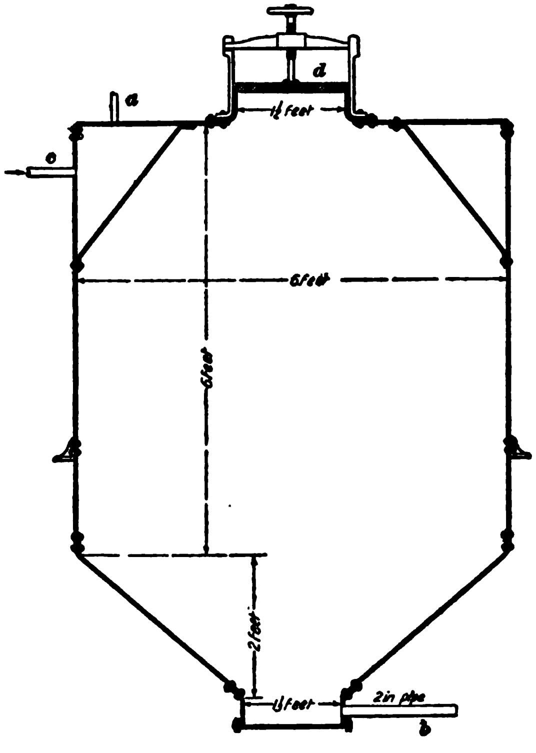

FIG. 10

59. In filtering the cylinder stocks, it is found that the first to come off have a very poor cold test and are of an almost white color. This is frequently used as a first-grade vaseline. Filtered cylinder stocks are always of a poorer cold test and viscosity than the unfiltered stocks from which they are made.

Many dealers prefer a filtered cylinder oil, but experienced engineers know that a dark, unfiltered cylinder oil is usually of much greater value as a lubricant than a light one. All that is necessary is to remove coke, suspended matter, and moisture, and all beyond that tends to reduce its value rather than improve it. The oil may gain in appearance at the expense of cold test and viscosity. This same rule will apply in the filtration of machine oils. Yet there are instances where a machinist may require an oil which is colorless and free from matter which would leave a stain.

Fullers' earth is being used very extensively in the filter house in place of bone black. Very frequently, illuminating oils are filtered through long filters of this earth, to change an off-colored prime-white oil into a water-white.

60. Low-Grade Lubricating Oils. — Besides these higher grades of lubricating oils there are a number of black oils manufactured for the lubrication of railroad cars and heavy machinery, where efficiency and cheapness are the only requirements. These black oils are usually produced in the same manner as the cylinder stock mentioned previously. The best crude oil is never used for this purpose, but now generally the Ohio and Indiana sulphur oils are used.

The common products are known as summer and winter black oils of 25°, 15°, and 0° cold test. The difference between the summer and winter black oils is simply a matter of congealing point. Since these oils are nearly all sold in tank-car quantities at the refinery, the purchaser places his special requirements on the oil desired; as a result, what is known as summer black to one purchaser may be a winter black to another.

The black car oil commonly used on freight cars is a black oil of 25° cold test and about 29° Baumé gravity. The oil known as 21° grease stock is made from Ohio crude and run with bottom steam to 21° Baumé gravity. It is used as a base for the manufacture of black axle and roll grease, and as a paving tar to be mixed with asphalt in street paving.

61. Table IV gives some of the more common pure mineral-oil lubricants manufactured in a refinery.

TABLE IV

Class of Oil	Degrees Baumé	Flash, Degrees F.	Fire, Degrees F.	Viscosity
32 neutral	32	365	415	*183
28 pale paraffin	28	370	425	*185
26 red	26	375	435	*195
23½ red	23.5	510	570	*300
Filtered cylinder	26	535	595	*135
600 cylinder	26 to 28	540	600	†171
650 cylinder	25 to 27	585	650	†190
700 cylinder	24 to 25	610	690	†215
Reduced black oils:				
Summer black	29	350	390	
Winter black	29	335	370	
25°–30° F. cold test	29	350	390	
15° F. cold test	30	325	360	
0° F. cold test	30	315	350	

* Tagliabue instrument. † Saybolt instrument.

62. Advantages in the Use of Mineral Oils as Lubricants.—Few users of lubricants fully appreciate the value of a mineral lubricating oil in comparison with the vegetable and animal oils formerly used exclusively. The advancement in the manufacture of petroleum and its lubricating products within the last decade have been especially marked. The cause of the slow gain in popularity was due to the opposition that the mineral oils met with when it was first attempted to gain recognition for them as lubricants. This brought about methods of testing the real economic value of oils and processes to improve deficient qualities and remove or greatly modify objectionable features. These tests have also drawn a marked line of distinction between the empirical manufacturer of machinery oils and the one who is thoroughly familiar with the progress of scientific knowledge and the advances of manufacturing skill.

The modern lubricant manufacturer is desirous of knowing the particular purpose for which the oil is required, so that he can choose an oil, either pure mineral or compounded, which will give the best satisfaction for the purpose.

Mineral lubricants are used on all kinds of machinery; they are the safest and cheapest, and generally superior to animal and vegetable oils. They are safer on account of their high fire test, as they will resist great heat before vaporizing. They are more reliable, as they are usually pure, uniform in quality, and free from acid. The injurious effect of mineral oils on the metals of the bearings of any machine is less than one-half that of any pure vegetable or animal oil used as a lubricant.

Mineral lubricating oils are not affected by high-pressure steam or alkalies, while vegetable and animal oils are. The former last longer and work cleaner, they neither gum nor stain materials and manufacturers' products. It is not to be inferred from this, however, that animal or vegetable lubricating oils are driven from the market or are likely to be. They have distinct and important uses, and whether alone or combined with mineral oils, will continue to be employed for the purpose of lubrication.

63. Compounded Oils.—Although the pure animal and vegetable oils are falling somewhat into disuse, the compounded oils are becoming the principal lubricant for certain purposes; but it is impossible to lay down any fixed law that would enable an engineer to choose the right compound for any special machinery. The oils best suited for compounding with mineral oils are as follows:

VEGETABLE OILS	ANIMAL OILS
Rape-seed	Neatsfoot
Olive	Lard
Palm	Tallow
	Sperm
	Whale

These oils when used in small quantities do not readily gum or become rancid. They usually increase the viscosity and staying quality of the oil and when not used in excess do not increase the action on the metal. Castor and cottonseed oils are often used in compounds, but their use should not be encouraged, as they are far more liable to gum, and therefore retard rather than aid lubrication.

64. In the use of a lubricant, much depends on the intelligence of the engineer. He must know the speed, temperature, and work of his machine before he can form an idea of what oil would be best. Engineers have been known to work along using an oil either too light or too heavy for their work, without realizing that they could obtain an oil perfectly adequate in every respect for the same price or possibly less than that which they were paying. Cylinder oils are condemned as useless simply because engineers are using more oil than is necessary. Many engineers still hold that tallow is the only lubricant for steam cylinders and are constantly clamoring for a cylinder oil containing 25, 30, and even 50 per cent. of tallow oil.

Cylinder oils for low-pressure steam may be compounded with 5 to 10 per cent. of neatsfoot or tallow oil; but for high-pressure steam they should never contain more than 5 per cent. of animal oil. If the cylinder stock is strictly high grade, over 685° F. fire test, it has been found to give excellent satisfaction when entirely free from animal oil.

65. Effect of High-Pressure Steam on Animal Oils. Although the animal oil may lubricate a steam cylinder seemingly with general satisfaction, one must bear in mind that with high-pressure steam there is a decomposition, which is injurious to the cylinder and piston. The decomposition of tallow, for instance, is to form glycerine and stearic acid. Hot stearic, palmitic, or oleic acids readily attack copper and its alloys, as well as steel, iron, etc., and therefore it is not difficult to see from the above how it is that steam cylinders and pistons are eaten away when a fatty oil, either by itself or in admixture with mineral oils, is used for lubricating them.

So great is the action, that there was removed from a 10″ × 12″ cylinder 2 pounds of a very hard, black substance that had been rolled into balls by the action of the piston. This substance contained 31 per cent. of fatty matter, 23 per cent. of moisture, and 46 per cent. of iron, and was the result of using a fatty oil lubricant. The piston and cylinder showed unmistakable proof of the action of the oil, although the latter had only been in use some 6 or 8 months, and the pressure of steam used in this case had not exceeded 40 pounds per square inch.

Instances similar to the above are of frequent occurrence about large manufacturing establishments, and a number of cases similar to this have come under the observation of the author where only a pure mineral oil would answer the purpose.

Heavy machine oils may be compounded with 10 per cent. of rape seed, olive, lard, or sperm oil.

Light machine oils are found to be better without any fatty oil.

Besides this class of compounded lubricating oils, there are oils compounded with soaps and solids for special purposes. In most cases they have been found very injurious to the machinery; while in a very few instances they are the most useful and valuable lubricants made today. The soaps commonly used are the aluminum and lead soaps. The addition of these soaps to a mineral oil increases the viscosity or body of the oil.

66. Mineral Castor Oil.—A very common oil made by the use of aluminum soap is a so-called *mineral castor oil.* It is an exceedingly viscous oil and may be used with a reasonable degree of satisfaction in some instances, yet in many places where it is used it would be better to use a pure mineral oil.

67. Galena Oils.—These oils, patented and first manufactured by Charles Miller, of Franklin, are compounded oils of a lead soap and Franklin heavy oil. It is probably the most noted compounded oil ever produced, its principal use being the lubrication of railway coaches.

68. *Graphite*, *soapstone*, and *mica* are used with mineral oils for special purposes. All these solids possess lubricating qualities within themselves, and when mixed with an oil become very efficient for the lubrication of excessively heavy and slow-running machinery.

69. Greases.—Greases having a semisolid to solid consistency are becoming a very important factor in the lubrication of certain kinds of machinery. Good greases are undoubtedly the best and most economical lubricants for machinery, if properly applied. The only drawback in their more general use is the difficulty experienced in applying them to certain bearings.

Greases are usually made from an insoluble soap mixed with a certain percentage of mineral oil. Insoluble soaps are those made from lime and rosin oil, cottonseed oil, or horse fat. They may be made solid or semisolid, according to the amount of mineral oil used in proportion to the soap. Under this head there are two classes of greases, *set* or *axle grease*, and *engine grease*. The former is made from a lime soap of rosin, mixed with a mineral oil. The latter is made from lime soap of horse fat or cottonseed oil, boiled with mineral oil. Both classes may be mixed with lead oxide, mica, or graphite.

70. Roll Grease.—Grease to be used on the rolls in the iron and tin-plate mills is frequently nothing other than wax tailings.

The list of compounded oils and greases is numberless; but their value is frequently not so much due to a particular percentage of composition as to the careful manipulation of the compounder.

THE MANUFACTURE OF VASELINE, WAXES, ETC.

71. Vaseline.—Vaseline was first introduced and patented by the Chesebrough Manufacturing Company, New York, and is, strictly speaking, a lubricating oil. It is made by the distillation of selected crude oil in the same manner as the cylinder stock already spoken of. The heavy cylinder stock, so to speak, is filtered through bone-black filters until the required color has been obtained; or the first filtration, which is of a light color, is used for vaseline, and the darker part used as filtered cylinder stock. The entire output of light cream-colored vaseline is bought up and controlled by the Chesebrough Manufacturing Company.

72. In order to bring the consistency and melting point to the proper degree, pure paraffin is melted and added to the filtered product. Its melting point is held at 86° F. or over. The United States Pharmacopœia gives the melting point at 104° to 125° F. Although the Chesebrough Manufacturing Company control the use of the name vaseline, other manufacturing concerns produce cosmolene, petrolene, and petroleum jellies, which differ only in name from vaseline.

The examination of Galician vaseline by Engler and Böhm is of considerable interest from a practical as well as from a scientific standpoint. The German Pharmacopœia considers a solution of paraffin in heavy oil as artificial vaseline. The examination of Engler and Böhm showed a marked difference between this imitation and the natural vaseline, a difference which is of interest to the commercial chemist, and one which can readily be made use of in the examination of a lubricating oil. The expansion on heating paraffin is much greater than that of vaseline. This difference in the coefficient of expansion is noticed in taking the gravity at different temperatures.

The natural vaseline shows very few crystalline bodies, where the artificial is quite crystalline; but on distillation the natural vaseline also becomes a mixture of crystalline paraffin and a heavy oil. The natural vaseline is thus shown to consist of a mixture of solid (not crystallizable) and liquid hydrocarbons of almost identical percentage of composition and boiling points. This latter, in connection with the expansion coefficient and viscosity, are the distinguishing features which make the natural vaseline a more desirable article than the artificial. As a lubricant, the artificial is almost worthless.

73. Besides manufacturing vaseline from the best crude oil, much of it is made from the salvy residues of old tanks and pipe lines known as *B. S.* of the oil industry. The same product is obtained from the tubing and rods of pumping wells, and is known as *rod wax*. This refuse material is put into a still and heated with fuel and superheated steam, or, in some cases, air. In this manner the light oils still remaining are blown off. The remaining salvy mass is filtered through bone black, when it produces a vaseline of high melting point and good consistency. Thousands of barrels are thus produced each year from material which previous to this had been one of the greatest nuisances with which the producer had to contend.

The value of vaseline to the drug industry can hardly be overestimated. Vaseline, having no action or chemical affinity for most of the drugs, salves may be compounded with it which remain unchanged for years. Animal and vegetable fats and oils have been displaced almost entirely by its use. The "American Druggist" considers the following vaselines official:

Petrolatum Liquidum, liquid at ordinary temperature.
Petrolatum Molle, melting point 104° to 113° F.
Petrolatum Spissum, melting point 113° to 125° F.

Liquid petrolatum and albolene, made by McKesson and Robbins, are heavy oils practically devoid of paraffin, and carefully filtered to remove all color and odor. Any heavy machine oil with good cold test may be prepared in this way.

74. Waxes.—If the rod wax mentioned above be pressed directly instead of being put into the still and reduced, it will yield a low melting solid of a salvy half-paraffin nature.

This was the first material used in making chewing gum from any of the petroleum products. At present nearly all chewing gums are made by dissolving a certain percentage of either Chicle or Balata gum in a low melting and poorly crystalline paraffin as a basis, and to this is added some flavor or drug from which it receives its name and particular value.

Much of the very cheap gum is made without the addition of any gum. The petroleum product used is a compound, the nature of which lies between that of vaseline and paraffin.

Vaseline, amorphous, melting point 86° F.

Chewing-gum wax, partly crystalline, melting point 116° F.

Paraffin, crystalline, melting point 125° F.

PETROLEUM AND PRODUCTS

(PART 3)

TESTING OF PETROLEUM

1. Introductory.—These tests have reference to certain physical and chemical properties that the products of petroleum are required to possess in order to be considered of value in particular uses. In practice, as a rule, an examination of the physical properties is considered sufficient for the determination of the value of an oil; however, certain chemical properties are of no less importance in determining the real character of the oil. Most of these tests are carried out by means of especially constructed apparatus.

2. Specific Gravity.—The determination of the specific gravity is one of the tests that is considered important in every product from the lightest to the heaviest. It is considered important in the lighter products, since volatility is directly proportional to the degrees Baumé. In America the specific gravity is usually determined by means of hydrometers with the Baumé scale.

A weighing jar *A*, Fig. 1, is four-fifths filled with the oil to be examined; a hydrometer *B*, with a thermometer in the lower part, is introduced, and the depth to which the instrument sinks read off. In order to obtain accurate results, the instruments should be of the large type and graduated to $\frac{1}{10}$ degree. In practice, corrections for temperature are made by means of Tagliabue's "Manual for Inspectors of Coal Oil." These tables, however, are not perfectly reliable

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for light oils, and in cases where close results are required, it is best to weigh at 60° F. The specific gravity at 15.5° C. may be found by the formula

$$\text{Sp. Gr.} = \frac{140}{130 + \text{Baumé}}.$$

3. Testing of Gasoline or Benzine.—In the purchase or sale of gasoline and the light essences, the gravity is usually the only test that is made. It is a well-known fact to the refiner that the gravity requirement is of little consequence if he desires to produce a cheaper article. The free evaporating gasoline is what is wanted, and must be produced by avoiding all heavy naphtha. For instance, a stove gasoline of 70° Baumé, made from a cut between 74° and 66° Baumé, containing 9° in the cut, must be better than a gasoline made from lighter oil cut from 80° to 60° Baumé, containing 20° in the cut. A light crude will produce a better gasoline than a heavy oil, if the refiner will give the customer the benefit of a lighter product; but if he endeavors to increase his percentage and keep the gravity down, the consumer must suffer. The only test that will determine this evil is the distillation test, which is carried out in a similar manner to the distillation of the kerosene. Besides this, the gasoline must be odorless and free from acid. For boiling points, the student is referred to Table I.

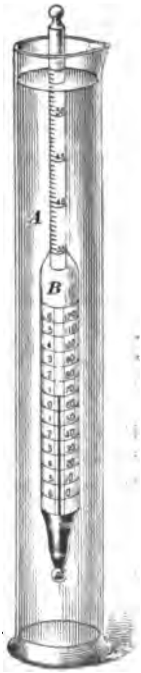


FIG. 1

4. Odor and Acidity Test.—In order to test the odor of gasoline, take a piece of cloth, saturate it with the gasoline, and partially evaporate, when any foreign odor will become distinctive. The acid test is the same as that given under kerosene. Benzine to be used for pharmaceutical purposes, or for the mixing of paints and varnishes, is required to be odorless, free from acid and oil, and from 60° to 63° Baumé. The test of the odor is taken similar to that of the gasoline.

5. Test of Benzine for Oil.—To test for oil, dip a sheet of writing paper into the benzine so as to cover about half the sheet, and permit it to evaporate. After evaporation, it should be perfectly free from any signs of grease and should have the same appearance when held towards the light as the part that was not saturated.

Another test is to burn a little of the benzine in a small evaporating dish and observe the amount of soot deposited on the sides of the dish and the color of the flame. If there is any soot after the burning, or a red-colored flame, it is a sure indication of oil in the benzine. No odor should be emitted while burning. The specific gravity is taken as usual.

6. Distillation Test for Benzine and Burning Oils. The same cautions regarding long cuts are true of benzine as well as gasoline, and the only means for determining this fact is by the distillation test. A good 63° Baumé deodorized benzine should distil between 248° and 302° F. The smallest fraction, if any, must pass over above 302° F. By referring to Table I, the approximate boiling points will be seen. The distillation test is the best test that can be applied to a burning oil in determining its value as an illuminant.

The amount of light oils is limited by the fire-test requirements, and the heavy oils, which are poorer illuminants in an ordinary lamp, must be determined by Engler's test, previously described. Instead of making a distillation of one fraction, as described in the test for the burning oil, it is well to divide the distillate into as small fractions as possible (every 50° F.). In a first-class illuminating oil, these fractions will be very nearly of equal volume.

It is not sufficient to know that an oil distils between 302° and 572° F., but also the percentage of the intermediate degrees should be known. For instance, an oil may be 48° Baumé, 150° F. fire test, and distil between 302° and

572° F. and yet be a very poor illuminant, on account of the cut at the still. It is possible to make an oil of a light fraction mixed with a heavy fraction that will have a good fire test and gravity and yet fail to make a free burning oil. An oil of that nature may burn well for a time and then refuse to burn on account of the heavy oil in its composition. The distillation will always reveal the manner in which the gravity and fire tests were made. The best kerosene is always made of the entire cut of the burning oil as it comes from the crude still. Much of the export oil is made from the cuts above and below the best water white. This is not only known to the American refiner, but has been distinctly shown by Engler in his distillation test.

7. The following are the results of tests on three different oils as given by Engler and C. Schestopal:

Alsace Brilliant petroleum.......	.801 Sp. Gr.
Kaiseroel (Imperial oil)..........	.795 Sp. Gr.
Ordinary Pennsylvania petroleum.	.800 Sp. Gr.

One hundred cubic centimeters of each were distilled, according to Engler's method. The results of these tests are given in Table I.

This does not mean that the Pennsylvania petroleum is inferior to the German oil, but that the American refiner has exported tops and bottoms and used the heart of his burning oil for some special oil to be consumed at home. The distillation is found to go beyond the limits, both above and below (302° to 572° F.). The light oil is not found to be injurious to the burning quality, but it injures the fire test. These distillation tests are coming into common use for the lighter as well as the heavier oils.

8. Flashing Point and Fire Test.—Petroleum consists of a mixture of hydrocarbons, and with a preponderance of light, volatile oils, its use is dangerous. When the readily volatile oils are not sufficiently separated by distillation, even at the ordinary temperature of a room, kerosene frequently evolves vapors that form, with the air in the fount of the lamp, explosive gas mixtures. According to Chandler's experiments, the most violent explosion occurs with a mixture of 1 part petroleum vapor and 8 or 9 parts air; 1 part air and 3 parts petroleum vapor give a slight report, while a mixture of 1 part air and 1 part petroleum vapor does not explode.

To what degree a small percentage of naphtha will reduce the degree of temperature at which the oil evolves inflammable vapors is shown by the following experiments of Dr. C. B. White, of New Orleans, Louisiana. With an oil that originally yielded inflammable gases at 113° F., the respective degrees of temperature fell with the addition of 1 per cent. of naphtha to 103° F.; with the addition of 2 per cent. of naphtha, to 92° F.; with the addition of 5 per cent. of naphtha, to 83° F.; with the addition of 10 per cent., to 59° F.; with the addition of 20 per cent. of naphtha, to 40° F. The point at which these vapors will continue to burn when tested with a flame is called the *burning point*, or *fire test*. The *flashing point*, or point at which explosive vapors will rise, and the fire test can never coincide.

TABLE I

Varieties of Oil	Boiling Point. Degrees F.	From Boiling Point to 302° F.	302°–347° F.	347°–392° F.	392°–437° F.	437°–482° F.	482°–527° F.	527°–572° F.	Above 527° F.	
Alsace Brilliant	296.6°	.8	10.5	20.00	24.70	20.00	15.4	4.8	3.80	cubic centimeters
			8.2	15.90	20.90	16.20	12.5	3.9		grams
Kaiseroel	212.0°		12.5	17.20	16.90	15.40	14.5	11.8	11.70	cubic centimeters
			9.6	13.30	13.50	12.50	12.0	9.6		grams
Pennsylvania kerosene	212.0°	15.8	10.0	12.00	8.25	11.00	7.8	9.0	26.15	cubic centimeters
		5.1	7.7	9.42	6.42	8.96	6.8	7.5		grams

In testing illuminating oils, the flashing test is the most important, since it is the explosive vapors in small quantities that cause most of the accidents. Moreover, an oil with high flashing point is sure to have a high fire test, while the reverse is not true. Nearly all our States and foreign countries have regulations requiring a minimum flashing test, and in some cases both flash tests and fire tests.

9. Instruments for the Determination of Flashing Point.—A large number of instruments of various constructions are in use for testing the flashing point of oil. The results of a flash test vary according to certain conditions, viz., to the size of the oil surface and the distance between the flame or electric spark and the surface of the oil, and whether the oil is kept in motion or not. The two principal divisions under the testers are the closed and the open cups. The latter is the cup originally used; but for burning oils it has been almost superseded by the closed type.

The law was almost entirely put to naught on account of the ease with which the operator can influence his test. J. B. Marvin, M.D., says in his annual report, in regard to his experience with the open tester: "It was found that both the flash point and the burning point could be varied at the will of the operator, through a limit of several degrees, if the inspector is incompetent or careless."

10. Elliott Closed Cup.—One of the closed testers receiving considerable recognition by the various States is the Elliott cup, shown in Fig. 2, usually known as the *New York State Board of Health Tester*. It consists of a copper oil cup *d* holding about 10 ounces of oil—the quantity usually contained in lamps—heated in a water bath by a small alcohol lamp *a* or Bunsen flame. The cup is provided with a glass cover *c*, carrying a thermometer *b*, and a hole for the insertion of the testing flame, a small wax taper or wood splint, which gives a flame about the size of a pea. The New York State Board of Health gives the following directions for the use of the cup:

11. Determination of Flashing Point by Means of the Elliott Closed Cup.—"Remove the oil cup and fill the water bath with cold water up to the rivet mark on the inside. Replace the oil cup and pour in enough oil to fill it to within $\frac{1}{8}$ inch of the flange, or projection, joining the cup and the vapor chamber above. Care must be taken that the oil does not flow over the flange. Remove all air bubbles with a piece of blotting paper. Place the cork in the center hole of the glass top through which the thermometer passes, until its bulb is covered by the oil. Alcohol should be used in the lamp for heating the water bath, and the wick should be carefully trimmed and adjusted to a small flame. The rate of heating should be about 2° per minute, and in no case exceed 3°.

"As a flash torch, a small gas jet $\frac{1}{4}$ inch in length should be employed. When gas is not at hand, employ a piece of waxed linen twine. The flame in this case, however, should be small. When the temperature of the oil has reached 85° F., the testings should commence. To this end insert the torch into the opening in the glass cover, passing it in at such an angle as to well clear the cover, and to a distance about half way between the oil and the cover. The motion should be steady and uniform, rapid, and without any pause. This should be repeated at every 2° rise of the thermometer

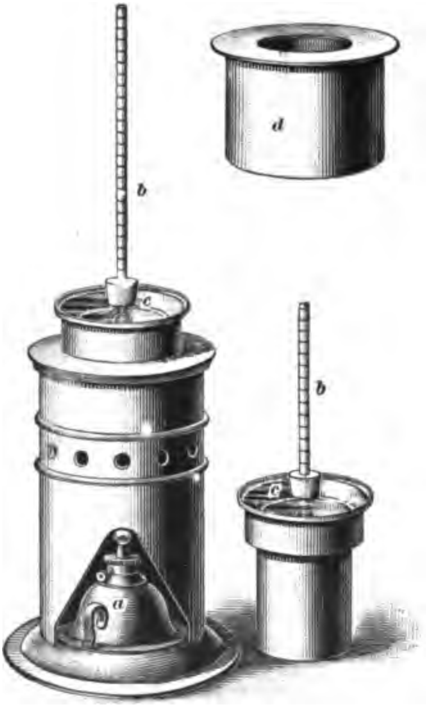


FIG. 2

until the temperature has reached 95°, when the lamp should be removed and a test made for each degree of temperature until 100° is reached. After this, the lamp may be replaced, if necessary, and the testings continued for each 2°.

"The appearance of a slight bluish flame, as seen through the glass, shows that the flashing point has been reached. In every case note the temperature of the oil before introducing the torch. The flame of the torch must not come in contact with the oil. The water bath should be filled with cold water for each separate test and the oil from a previous test carefully wiped from the oil cup."

12. Tagliabue Closed-Cup Tester.—This instrument, also known as Tagliabue's coal-oil pyrometer, is a closed cup of practically the same design as the foregoing, only it is a much smaller cup, and on that account is considered inferior to some of the other types.

13. Foster Automatic Oil Tester.—This instrument is generally conceded by oil inspectors to be the only American instrument where the personal element is left out of question. It is strictly automatic and results must be concordant. The instrument is shown in Fig. 3, and consists of a copper lamp furnace *a* containing a water bath and oil cup. The latter is surmounted by a closed vapor chamber, which is pierced at two points symmetrically placed for the reception of a thermometer *b* and a flashing lamp, or taper *c*; the apparatus being elliptical in shape, the thermometer is placed in one focus of the ellipse and the flashing taper in the other.

The flashing taper consists of a small cylindrical wick holder, supported by radial arms to an annular ring, and rests upon a similar ring at the bottom of an open, shallow basin, the spaces between the radial arms giving egress to the oil vapor, while the wick itself extends down into the body of the oil within the cup. An inverted conical thimble, resting upon the rim of the basin, prevents the dissipation of the vapor. The thermometer is mounted in a copper tube, cut away in front to expose the scale; the bulb of the thermometer when in position is within the body of the oil at a definite distance below the surface. An orifice around the tube of the thermometer, definite in diameter and distance above the surface of the oil, allows of a downward current of atmospheric air when the flashing taper is alight.

An index is placed within the water bath and the oil cup for maintaining uniformity in the filling of each. The heating lamp *d* of the lamp furnace has its wick adjustable, to facilitate uniformity in the rate of heating. All parts of the apparatus are definite and uniform in shape, size, and position. Each thermometer is of ascertained accuracy, and if instructions for using are carefully followed, the most accurate results are attainable.

14. Determination of Flashing Point by Means of the Foster Automatic Tester.—In determining the flashing point by means of the Foster automatic tester, proceed as follows:

1. Remove the thermometer with its mounting from the oil cup.

2. Lift off the oil cup containing the flashing taper and fill the open water bath with water, to the mark upon the inside.

3. Now take out the wick holder from the oil cup and fill this vessel with the oil (at 60° F.) to be tested, pouring in the oil at the place of the wick holder and noting the gauge mark at the thermometer hole; pour in the oil very gradually as soon as the surface of it approaches the gauge mark. The gauge mark consists of a small pendent shelf, and the oil cup is properly filled when the upper surface of the oil just adheres to the lower surface of the gauge mark.

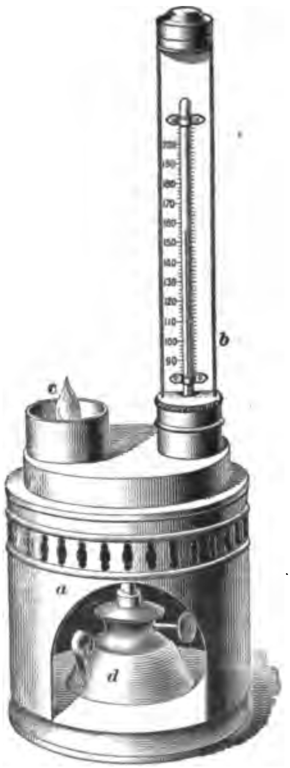


FIG. 3

Too much care cannot be taken at this point; therefore, after having ceased pouring, tip the cup so that the oil flows away from the gauge, and then, gradually restoring it to the horizontal, see that the surface again adheres and add a little more oil if it does not.

4. See that the wick of the flashing taper be adjusted to give a very small flame—a flame that does not exceed ¼ inch in height. A flame that exhibits as much blue at its base as yellow at its top is proper.

5. Now set the oil cup on top and into the water bath; return the flashing taper to its place, inverting the conical thimble around it, and return the thermometer to its place upon the cup; in doing this, be sure that the casing of the latter is pushed down upon the cup as far as it will go.

6. Fill the lamp beneath half full of alcohol, light it, and put it in its place beneath the water bath. Now note the rate of increase in temperature as shown by the thermometer, and adjust the wick to raise the temperature at the rate of 2° per minute. When the temperature has reached 100°, light the flashing taper and observe it closely. As soon as the oil under test has reached its flashing point, the flame of this taper will be extinguished by the first flash, and the point of attention is to note the temperature at the instant the flame of the taper is extinguished. This *flashing point* is the point of temperature at which the oil generates a vapor, the composition and character of which is that of ordinary illuminating gas. The *flash* indicates that this has formed an explosive mixture with atmospheric air.

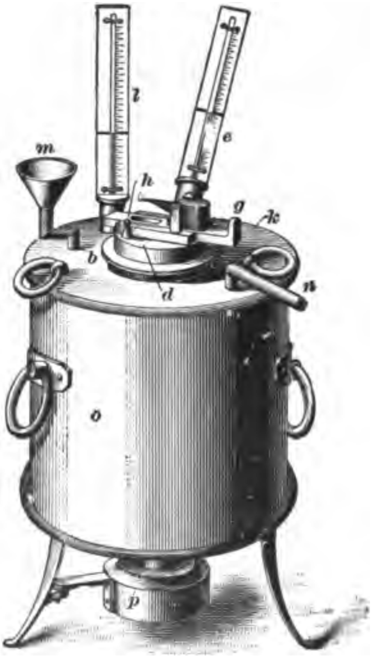
15. Determination of Fire Test by Means of the Foster Automatic Tester.—The fire test is made by continuing to heat the oil (the cover being removed in the case of a closed tester) at the same rate after the flash test is made and flashing the oil every 4° until the flame remains. The fire test of illuminating oils is usually from 15° to 25° F. higher than the flash test of the open cup.

In testing 300° oil—*Mineral Seal* or *Miners' Sperm*—the test is usually made in an open cup of brass. The oil is heated at the rate of 5° per minute and the test flame applied every 5° after 230° F. has been reached. The flash point should not be under 250° F. in the open-cup test. By comparing these cups and looking over the requirements of the different States, it will be seen that the refiners' sales of illuminating oils are of great variety. Each refiner is compelled to have on hand a number of reliable testers, but as these cups are not used regularly by the official oil inspector, the latter's tests in many cases will be unreliable. Every refiner has striven to reduce his tests to one instrument, using the one in which the error from the operator's use is reduced to a minimum. Tables II, III, and IV are attempts to give some comparison of the different testers.

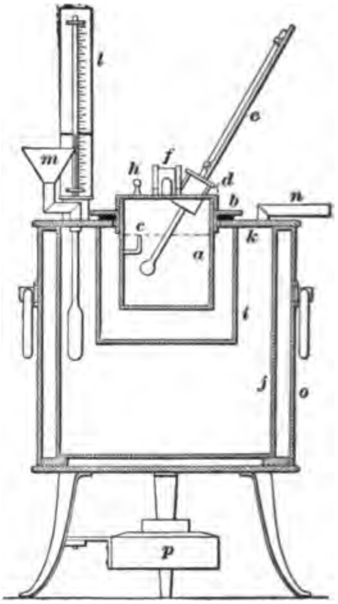
16. Abel Closed-Cup Oil Tester.—The apparatus shown in Fig. 4 (*a*) and (*b*), adopted by England, Germany, and Austria, is strongly recommended, as it is of importance to the American refiner and exporter, all export oil being tested by it. The instrument and its uses are thus described in the Schedule of the 1879 Petroleum Act of the British Parliament.

The oil cup *a*, Fig. 4 (*b*), consists of a cylindrical vessel 2 inches in diameter, $2\frac{2}{10}$ inches high (internally), with outward projecting rim *b*, $\frac{5}{10}$ inch wide, $\frac{3}{8}$ inch from the top, and $1\frac{7}{8}$ inches from the bottom of the cup. It is made of gun metal or brass. A bracket *c*, consisting of a short, stout piece of wire bent upwards and terminating in a point, is fixed to the inside of the cup, to serve as a gauge. The distance of the point from the bottom of the cup is $1\frac{1}{2}$ inches. The cup is provided with a close-fitting, overlapping cover *d* made of brass, which carries the thermometer *e* and test lamp *f*. The latter is suspended by two supports from the side by means of trunnions, upon which it may be made to oscillate; it is provided with a spout, or wick tube, the mouth of which is $\frac{1}{16}$ inch in diameter. The socket to hold the thermometer is fixed at such an angle and its length is so adjusted that the bulb of the thermometer, when inserted to full depth, shall be $1\frac{1}{2}$ inches below the center of the lid.

The cover is provided with three rectangular holes: one in the center, $\frac{5}{10}$ inch by $\frac{4}{10}$ inch, and two smaller ones (not shown in the figure), $\frac{3}{10}$ inch by $\frac{2}{10}$ inch, close to the sides and opposite each other. These three holes may be closed and uncovered by means of a slide *g* moving in grooves, and having perforations corresponding to those on the lid. In moving the slide so as to uncover the holes, the oscillating lamp is caught by a pin fixed in the slide and tilted in such an angle as to bring the end of the spout just below the surface of the lid. Upon the slide being pushed back so as to cover the holes, the lamp returns to its original position. Upon the cover, in front of, and in line with, the mouth of the lamp, is fixed a white bead *h*, the dimensions of which represent the size of the test flame to be used. The bath, or heated vessel, consists of two flat-bottomed copper cylinders, an inner one *i*, 3 inches in diameter and



(a)
FIG. 4



(b)
FIG. 4

2½ inches in height, and an outer one *j*, 5¼ inches in diameter and 5¾ inches in height. They are soldered to a circular copper plate *k*, perforated in the center, which forms the top of the bath, in such a manner as to enclose the space between the two cylinders, but leaving access to the inner cylinder. The top of the bath projects both outwards and inwards about ⅜ inch, that is, its diameter is about ⅜ inch greater than that of the body of the bath, while the diameter of the circular opening in the center is about the same amount less than that of the inner copper cylinder. To the inner projection of the top is fastened, by six small screws, a flat ring of ebonite, to avoid metallic contact between the bath and the oil cup. The exact distance between the sides and the bottom of the bath and of the oil cup is ½ inch. A split socket, similar to that on the cover of the oil cup, but set at a right angle, allows a thermometer *l* to be inserted into the space between the two cylinders. The bath is further provided with a funnel *m*, an overflow pipe *n*, and two loop handles. The bath rests upon a cast-iron tripod stand, to the ring of which is attached a copper cylinder, or jacket *o*, flanged at the top and of such dimensions that the bath, while firmly resting on the iron ring, just touches with its projecting top the inward turned flange. The diameter of this outer jacket is 6½ inches. One of the three legs of the stand serves as a support for the spirit lamp *p* attached to it by means of a small swing bracket. The distance of the wick holder from the bottom of the bath is 1 inch.

Two thermometers are provided with the apparatus, one *l* for ascertaining the temperature of the bath, and the other *e* for determining the flashing point. The thermometer *l* for ascertaining the temperature of the water has a long bulb and a space at the top. Its range is from about 90° to 190° F. The thermometer is adjusted with a metal collar fitting the socket, and the part of the tube below the scale should have a length of about 3½ inches, measured from the lower end of the scale to the bulb. The thermometer *e* for ascertaining the temperature of the oil is fitted with collar and ivory scale in a similar manner to the one described. It has a round bulb, a space at the top, and ranges from about 55° to 150° F.; it measures from end of ivory back to bulb 2¼ inches.

17. Determination of Flashing Point by Means of the Abel Tester.—The test apparatus should not be placed for use in a position where it is exposed to currents of air, or drafts. The heating vessel, or water bath, is filled by pouring water into the funnel until it begins to flow out at the spout of the vessel. The temperature of the water at the commencement of the test is to be 130° F. When a test has been completed, this water bath is again raised to 130° by placing the lamp underneath, which is done while the petroleum cup is being emptied, cooled, and refilled with a fresh sample to be tested. The lamp is then turned on its swivel from under the apparatus and the next test is proceeded with.

The test lamp is prepared for use by fitting it with a flat-plaited candle wick and filling it with colza or rape oil up to the lower edge of the opening of the spout or wick tube. The lamp is so trimmed that when lighted it gives a flame about .15 inch in diameter, and this size of flame, which is represented by the projecting white bead on the cover of the oil cup, is readily maintained by simple manipulation from time to time with a small wire trimmer. When gas is available, it may be conveniently used in place of the little oil lamp, and for this purpose a little test-flame arrangement for use with gas may be substituted for the lamp.

The bath having been raised to the proper temperature, the oil to be tested is poured into the petroleum very slowly until the level of the liquid just reaches the point of the gauge fixed in the cup. In warm weather, the temperature of the room in which the samples to be tested are kept should be observed in the first instance, and if it exceeds 65° F., the samples to be tested should be cooled down (to about 60°) by immersing the bottles containing them in ice water. The lid of the cup, with the slide closed, is then put on and the cup is placed into the bath, or heating vessel.

The thermometer in the lid of the cup has been adjusted so as to have its bulb just immersed in the liquid and must not be altered. When the cup has been placed in the proper position, the scale of the thermometer faces the operator.

The test lamp is then placed in position upon the lid of the cup, the lead line, or pendulum (the pendulum should be 24 inches in length from the point of suspension to the center of gravity of the weight), which has been fixed in a convenient position in front of the operator, is set in motion and the rise of the thermometer in the petroleum cup is watched. When the temperature has reached about 66° F., the operation of testing is to be commenced, the test flame being applied once for every rise of 1° in the following manner: The slide is slowly drawn open while the pendulum performs three oscillations, and is closed during the fourth oscillation.

Note.—If it is desired to employ the test apparatus to determine the flashing points of oils of very low volatility, the mode of proceeding is to be modified as follows: The air chamber that surrounds the cup is filled with cold water to a depth of $1\frac{1}{2}$ inches, and the heating vessel, or water bath, is filled as usual, but also with cold water. The lamp is then placed under the apparatus and kept there during the entire operation. If a heavy oil is being dealt with, the operation may be commenced with water previously heated to 120° F., instead of with cold water.

18. Tagliabue Open-Cup Tester.—Under the open testers, Tagliabue's open cup, shown in Fig. 5, is the most important. It was the official testing apparatus in England and many of the United States, but, as has been previously stated, has been superseded almost entirely by the closed type. It is similar in construction to the closed cups, having a brass water bath *a* and a glass oil cup *b*, but no cover.

19. Determination of Flashing Point by Means of Tagliabue's Open-Cup Tester.—The water bath is nearly filled with cold water, allowing for the displacement by the oil cup; the latter is filled to within $\frac{3}{32}$ inch of the top, care being taken not to wet the rim, and the thermometer placed in position. The heating flame is adjusted so that it is $\frac{3}{8}$ inch high, and the heating proceeded with at the rate of

$2\frac{1}{2}°$ per minute, until 97° F. is reached, when the test flame is applied and testings made every 3° until the flash point is reached. The test flame should be that of a waxed linen twine, wood splint, or gas jet. It is slowly and carefully passed over the kerosene about $\frac{1}{8}$ inch from the surface. The test depends very much on the position and size of the flame.

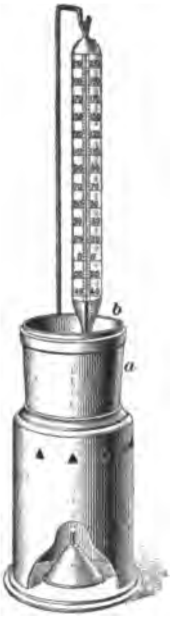


FIG. 5

Tables II, III, and IV show a comparison of the different results obtained with different testers.

20. Color Test for Illuminating Oils. The color test of illuminating oil is usually carried out in a 4-ounce sample bottle in which the oil is compared with standard colors prepared by means of solutions of potassium chromate in water. In America, the colors in use are: (1) Water white, (2) prime white, (3) standard white. In England (i. e., receiving ports): (1) Water white, (2) prime white, (3) standard white, (4) merchantable. In Germany (i. e., receiving ports): (1) Water white, (2) prime white, (3) standard white, (4) prime light straw to white, (5) prime light straw to standard, (6) light straw, (7) straw. The colors in the foreign countries are usually determined by some colorimetric apparatus.

21. Cloud Test.—This test is made as follows: Two ounces of the oil are placed in a 4-ounce sample bottle, with a thermometer suspended in the oil. The bottle is exposed to a freezing mixture of ice and salt, and the oil stirred with the thermometer while cooling. The temperature at which the cloud forms, due to crystallization of paraffin, is taken as the cloud test. Aside from the most common tests previously mentioned, it is very often necessary to make certain tests in regard to the purity of the oils.

22. Preliminary Acid Test.—Shake equal quantities of hot water and oil in a test bottle, pour off the oil, and test the water with litmus solution. The acid in this case is usually sulphuric, coming from the imperfect refining.

TABLE II

COMPARISON OF COMMON TESTERS

Foster Closed-Cup Flash. Degrees F.	Tagliabue's Closed-Cup Flash. Degrees F.	Elliott Closed-Cup Flash. Degrees F.	Tagliabue's Open-Cup Flash. Degrees F.	Tagliabue's Open-Cup Fire. Degrees F.	Grade of Oil
85	72	67	90	100	
90	77	70	100	110	110° oil
94	82	76	110	117	120° oil
100	89	82	109	125	
106	97	87	112	130	130° oil
111	100	94	115	135	135° oil
115	107	100	120	140	140° oil
120	115	108	124	147	150° oil
133	129	117	135	159	160° oil
146	142	130	154	170	175° oil

23. Qualitative Test for Sulphur.—In testing for sulphur in the imperfectly refined oils of Ohio, it is customary to treat a small sample with sodium plumbate. Any change of color indicates sulphur. Other compounds of sulphur may be detected by heating the oil to its boiling point with a bright piece of sodium or potassium. If sulphur compounds be present, a yellowish layer is formed upon the metal. After cooling, add distilled water, drop by drop, until the metal is dissolved, and test for sulphides with sodium nitroprusside. A fine violet color indicates sulphur. Aufrecht makes the following statement: "Good petroleum should not contain more than 10 to 15 milligrams of sulphur in 100 cubic centimeters."

24. Quantitative Estimation of Sulphur in Petroleum.—To 50 cubic centimeters of the petroleum, .5 gram

TABLE III

COMPARISON OF TESTERS (ENGLER AND HASS)

Name of Tester	Sample A				Sample B				Sample C			
	Number of Tests	Results. ° C.	Mean. ° C.	Difference. ° C.	Number of Tests	Results. ° C.	Mean. ° C.	Difference. ° C.	Number of Tests	Results. ° C.	Mean. ° C.	Difference. ° C.
Tagliabue open..	1	22.7 to 38.8	30.95	16.1	9	32.2 to 48.8	42.00	16.6	6	45.5 to 57.2	52.20	13.3
Tagliabue closed.					18	24.0 to 39.4	31.68	15.4				
Danish..........	5	19.5 to 21	20.80	3.5	4	29 to 31	30.00	2.0	4	42 to 45	43.25	3.0
Saybolt..........	4	36.6 to 36.7	36.30	1.1	2	36.1 to 36.6	36.35	.5	2	48.5 to 52.7	50.75	3.9
Parrish..........	5	20.7 to 23	21.40	2.7	15	25.5 to 30.7	27.30	5.2	9	36.5 to 39	37.70	2.5
Abel............	4	16 to 17.1	16.60	1.1	7	22.2 to 23.8	22.64	1.6	3	32.4 to 33.8	32.96	1.8
Engler..........	4	21 to 22.5	21.95	1.5	19	28 to 30.5	29.40	2.5	2	39.3 to 39.7	39.50	.4

TABLE IV

(REDWOOD)

Oil Sample	Tagliabue Open Cup Degrees F.	Arnabaldi Open Cup. Degrees F.	Saybolt. Degrees F.	Tagliabue Closed Cup. Degrees F.	Tagliabue Closed Cup. Large Size. Degrees F.	Wisconsin. Degrees F	Abel Closed Cup. Degrees F.	Benstein. Degrees F.	Millsp. Degrees F.	Mann. Degrees F.	Foster Automatic Closed Cup. Degrees F.
1	110	118	120	111	117	107	103	130	111	95	119
2	111	121	124	115	118	107	102	128	107	96	
3	119	122	122	112	118	108	102	130	108	95	118
4	97	96	97	90	93	86	76	90	81	75	96

of sodium bicarbonate is added, and the mixture is distilled at the rate of 40 to 50 drops a minute until about 45 cubic centimeters is distilled over. The residue is placed in a large porcelain dish and repeatedly washed with ether; after spontaneous evaporation of the latter, about .5 gram of sodium is added in small pieces and the whole evaporated over a small flame to a sirup, and then ignited with the gradual addition of ammonium nitrate until the ash is quite white. The residue is extracted by very dilute hydrochloric acid and the sulphuric acid estimated in the filtrate in the usual manner by barium chloride.

25. Sulphuric-Acid Refining Test. — The object of this test is to judge the degree of refinement to which the oil has been subjected, a well-refined oil giving little or no color when submitted to this process. A glass-stoppered test bottle is filled half full of the oil and about half as much chemically pure sulphuric acid of 66° Baumé added. This mixture is then shaken for several minutes. The depth of color indicates the impurities still remaining in the oil. In comparative tests this color is matched by various standard solutions of Bismarck brown.

26. Alkali Refining Test.—In order to reduce expenses, attempts are frequently made to employ less alkali than really is required. Although the oil shows a good color for a short time, this disappears after a little time and its illuminating properties are impaired. The test is made similar to that of the sulphuric-acid test, using a caustic-soda solution of about 22° Baumé and observing the change of color.

THE TESTING OF LUBRICATING OILS

27. Cleveland Open-Cup Fire Tester.—In testing lubricating oils, the open cup is used almost exclusively. The one shown in Fig. 6 is known as the *Cleveland open-cup fire tester*, and is commonly designated by purchasers of lubricants as the one to be used in making the flash tests and fire tests of the oils. It is made of heavy brass throughout with a solid glass thermometer. The cup *a* is filled with oil to within ⅜ inch of the flange, and in case of cylinder oils, to ½ inch. The flange is carefully dried with blotting paper. The thermometer *b* is so placed as to have the bulb completely covered with oil. The gas flame is regulated so as to heat the oil at the rate of 15° F. per minute. The testing flame should be first applied at 50° F. below the supposed flash test, and then every half minute until the flash point is reached. In making the fire test, heating is continued and the flash test made every half minute until it remains burning.

28. Specific Gravity.—The specific gravity of cylinder oils is taken in a manner similar to that of illuminating oils, but must be at a higher temperature and the reduction made according to Tagliabue's "Manual for Inspectors of Coal Oil."

29. Viscosity Tests.—The viscosity of a lubricant is usually considered its most valuable quality. This is usually taken by means of the apparatus called a *viscosimeter* (see *Quantitative Analysis*). A great variety of these have been constructed, all based upon the length of time required for a given quantity of oil to flow through a small orifice, as compared with water or some other oil used as a basis. The best-known instruments are those of Saybolt, Tagliabue, and Engler.

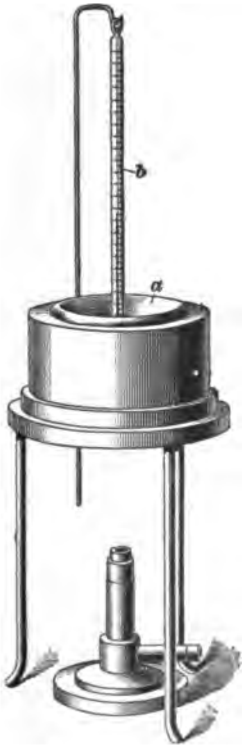


FIG. 6

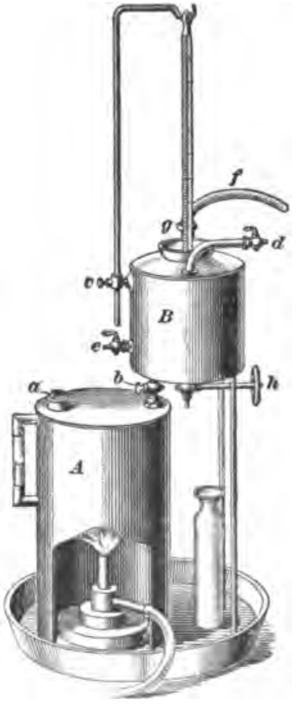


FIG. 7

30. Saybolt Viscosimeter.—This instrument, which is under the control of the Standard Oil Company, is made in three forms—A, B, and C. Apparatus A is the standard for testing distillates at 70° F.; B, for testing the light, black oils at 70° F.; and C, for testing heavy, black oils and cylinder oils at 212° F.

31. Tagliabue Improved Viscosimeter.—The Tagliabue improved viscosimeter, shown in Fig. 7, is generally used by the independent refiners. The instrument as shown consists of two metallic tanks, or receptacles *A* and *B*. The lower one *A* is a water boiler, while *B* is a vessel in which is placed the receptacle containing the oil to be tested. Direct communication is established between *A* and *B* by means of the stop-cock *b*. The stop-cocks *d* and *e* and the coupling *g* communicate with the interior of *B*. The stop-cock *h* communicates with the oil receptacle, so that the oil may flow out through nipples of specified sizes attached to it. The thermometer is suspended in the oil as shown. When testing at 212° F., water in *A* is boiled and steam passes through the stop-cock *b* into *B*, thus warming the oil in the receptacle to the required temperature. When testing at lower temperatures, *b* is closed and water heated to the required temperature is poured into *B*.

32. Test for the Viscosity of Oils at 212° F.—Pour water into boiler *A* through opening *a*, after unscrewing the safety valve, until the water gauge shows that the boiler is full. See that stop-cock *b* is open, making direct connection between the boiler and the upper vessel that surrounds the receptacle in which the oil to be tested is placed. Place a wire holder in the set nut *c* and suspend a thermometer so that its bulb will be about ¼ inch from the bottom of the oil bath. Then, after carefully straining 70 cubic centimeters of the oil to be tested, which, of course, must be warmed in the case of very heavy oils, pour the same into the oil bath. Close stop-cocks *d* and *e*. Screw the extension *f*, with rubber hose attached, into coupling *g* and let the open end of the hose be immersed in a vessel of water, which will prevent too large a loss of steam. Place a lamp or a Bunsen burner under the boiler, screw the steel nipple, furnished with the apparatus and marked 212, on to stop-cock *h*, and the apparatus is ready to use.

After steam is generated, wait until the thermometer in the oil bath shows a temperature of from 209° to 211° F.; then place the 50-cubic-centimeter test glass under stop-cock *h* so that the stream of oil strikes the side of test glass, thereby preventing the formation of air bubbles, and, when the thermometer indicates its highest point, open the cock *h* simultaneously with the starting of the watch, which is supplied with each instrument. When the running oil reaches the 50-cubic-centimeter mark in the neck of the test glass, the watch is instantly stopped and the number of seconds noted. Then multiply the number of seconds by two, and the result will be the viscosity of the oil. For example, if 50 cubic centimeters of oil runs through in 101½ seconds, the viscosity will be 203. It is necessary to keep the oil well stirred before making the test, in order to have the oil at a uniform temperature.

33. Test for the Viscosity of Oils at 70° F.—Screw steel nipple, furnished with the apparatus and marked 70, on to faucet *h* and close stop-cock *b*, closing communication between the boiler and the upper vessel; also close stop-cock *e*. Fill the upper vessel through opening *g* with water at a temperature as near 70° F. as possible; also have the oil to be tested at same temperature. Hang the thermometer in position, and after stirring the oil thoroughly, blow through rubber tube at *d*, to thoroughly mix the water. Should the thermometer show higher or lower than 70°, add cool or warm water until the desired temperature is attained. Then proceed as before.

NOTE.—In making tests at 70° F., it is absolutely necessary that the temperature of the oil should be fully 70° before starting the run.

34. Engler Viscosimeter.—This instrument is commonly used in Europe, and although the viscosity numbers differ from the foregoing, it is constructed on a similar principle.

35. Cold Test.—The cold test of an oil is that point at which it will not flow freely from a vessel. In order to make this test, put about 1 ounce of the liquid to be tested into a common 4-ounce sample bottle and place a short, stout thermometer in it. Then place the bottle in a refrigerator, where the liquid will become frozen. When the liquid has become solid throughout, remove from the refrigerator and allow the liquid to soften, thoroughly stirring and mixing it at the same time, by means of the thermometer, until the mass will run from one end of the bottle to the other. Now grasp the bottle by the neck, having in the same hand a little waste or a towel, which encloses the thermometer, withdraw the thermometer through the waste, to wipe it far enough to see the mercury, and read the temperature. The reading is the cold test of the liquid.

36. Chilling Point.—The chilling-point test is employed where it is desired to know whether the liquid remains clear at any given temperature. Use a similar bottle and thermometer and the same amount of liquid as for cold test. Expose the liquid to the given temperature, or preferably a little below; stir it with the thermometer occasionally, until the temperature of the whole liquid becomes the one desired, and then allow it to stand for the time specified; at the end of which observe whether the liquid remains transparent and free from flocks and scales of congealed constituents. If so, it stands the test, or chilling point.

37. If it becomes necessary to know at what temperature flocks, or scales, of congealed constituents, or more or less opacity actually takes place, proceed as follows: Use the same kind of bottle and thermometer and the same amount of oil as for the cold test. Expose the oil to a temperature 5° colder than that at which the liquid remained perfectly clear, that is, free from flocks and scales, and allow the temperature to fall below that, with constant watching and occasional stirring. Lower the source of cold 5° more and treat as before. Continue until opacity, or flocks or scales, begin to show. The reading of the thermometer when this point is reached is the temperature sought. It is well to determine a few of the intermediate points of solidification, such as the temperature, when it just begins to solidify, the temperature at which it will stick to the thermometer, like vaseline, and the temperature when it becomes hard, like butter.

38. Congealing Point.—For soap stock, the following test has been used: A 4-ounce sample bottle half filled with a sample of oil, with a thermometer inserted, is placed in a freezing mixture and chilled. The point at which the thermometer becomes stationary for some time before the mass becomes solid is considered the *congealing point*. This point is always higher than the cold test.

39. Freezing Mixtures.—Quite a number of methods have been proposed for obtaining the low temperature to be used in taking cold tests and chilling points. However, none of these methods is apparently so simple, so easily manipulated, and so expeditious as freezing mixtures. Three cases are to be considered:

1. *Mixtures for Cold Tests.*—If the cold test is above 35° F., use a mixture of snow or pounded ice and water in any suitable non-conducting vessel. A good-sized vessel and a plentiful supply of snow or pounded ice works best. The bottle with oil and thermometer is plunged into the freezing mixture and allowed to remain there until the liquid is frozen solid.

For cold tests from 35° F. down to zero, use a mixture of 2 parts, by weight, of snow or pounded ice and 1 part of common salt. The same remarks as above in regard to vessel, plentiful supply, and manipulation apply.

For cold tests from 0° F. to – 30°, use a mixture of 3 parts, by weight, of crystallized calcium chloride and 2 parts of snow or very finely pounded ice. The crystallized calcium chloride, if obtained in the market, should be crushed in a mortar into pieces not larger than wheat kernels. For cold tests approaching the lower range of the above limit, the vessels should be non-conducting and should be cooled to 32° F. before putting in the calcium chloride and ice. The crystallized calcium chloride may be bought in the market, but the following method gives a very cheap and efficient article from materials always at hand in every good laboratory, viz.:

Put about 1 pound of pulverized carbonate of lime (marble dust works best) into a casserole holding from 3 pints to 2 quarts, and pour concentrated commercial hydrochloric acid on it, a little at a time. A neutral solution of calcium chloride is formed very quickly, after each addition of acid, and in a short time the mass becomes wet enough to be stirred, to facilitate the action. Proceed with the addition of the acid until nearly all the marble dust is dissolved, taking care to avoid an excess of acid. Pouring the acid on to the marble dust, instead of adding the dust to the acid, prevents the suffocating fumes of HCl that are sure to be given off if the latter procedure is followed.

After the last addition of acid, filter into a convenient metal vessel whose weight is known; boil until a drop of the liquid placed on a watch glass and cooled by placing the glass on a lump of ice becomes solid rather quickly; then cool by placing the vessel in another vessel containing ice and water. As the temperature falls the calcium chloride should be stirred, to prevent the formation of a compact mass that is not easily broken up. If the operation is properly performed, when the temperature of the calcium chloride reaches 40° F. or a little below, the material in the metal vessel will be a more or less mushy mass of calcium-chloride crystals.

Now remove the metal vessel from the ice water, wipe it dry on the outside, and weigh, to determine the amount of calcium chloride. Wrap the bottom and sides of this vessel well with towels or other non-conducting material, and then for every 3 parts of calcium chloride add 2 parts of snow or finely pounded ice and stir thoroughly. After a minute or two, the material to be tested can be placed in the liquid, and the whole thing should then be covered, to prevent access of heat.

2. *Mixture for Cloud Tests.*—When it is desired to find out whether a liquid remains clear at any given temperature, the following is recommended:

(*a*) For temperatures above 32° F., place 1 gallon of water in a wooden bucket and regulate the temperature by adding either ice or cold water. Care should be taken to keep the water at the desired temperature while the liquid in the sample bottle is cooling.

(*b*) For temperatures from 32° F. down to zero, add to 1 gallon of water, in a bucket of wood or indurated fiber, 15 pounds of cracked ice. Stir thoroughly with a wooden stick, and when the temperature has reached 32° F. or thereabouts, add, with continued stirring, sufficient dry common salt to produce the temperature desired. Approximately, each quarter pound of salt added will lower the temperature under the conditions given 2° F., until 10° is reached, when twice the quantity must be added to bring the temperature down 2°.

As the usual specified temperatures at which chilling points are observed are 32°, 20°, 10°, and 0° F., it is fairly safe to say that these temperatures may be obtained as follows: For 32° F., into a wooden or indurated-fiber pail put 1 gallon of water and any convenient amount of ice, provided only sufficient is used; for 20° F., 1 gallon of water, 15 pounds of cracked ice, and 1½ pounds of dry common salt; for 0° F., ½ gallon of water, 15 pounds of cracked ice, and 5 pounds of dry common salt.

A little experience will enable the desired temperatures to be reasonably well controlled. Of course, the melting of the ice continually dilutes the salt solution, and if a fairly constant temperature for some time is desired, occasional small additions of salt will be necessary. For temperatures below 0° F., crystallized calcium chloride may be used in place of common salt. So much depends on the amounts of the materials used (especially when dealing with such low temperatures), on the appliances, and the protection given to the vessels in which the cold is produced that it is perhaps hardly wise to try to give proportions. It may be said, however, that 3 pounds of finely crushed ice and 2 pounds of crystallized calcium chloride in a properly cooled and protected vessel will give 10° below zero F., and 3 pounds of finely crushed ice and 3¾ pounds of crystallized calcium chloride under the same conditions will give 20° below zero F. A few experiments, however, are worth more than a good many directions.

3. *Mixtures for Chilling-Point Tests.*—When it is desired to know at what temperature flocks, or scales, of congealed constituents of more or less opacity take place, use the methods of obtaining desired temperatures described in the preceding paragraph, except that in going down the scale, temperatures 5° apart are produced by the proper mixtures and used as described.

40. Evaporation Test.—The object of this test is to determine what percentage of the oil is volatile when exposed to the conditions existing on the bearings to which the oil is to be applied. It may be carried out by means of a small quantity of oil on a watch glass, the whole first being accurately weighed. The watch glass with oil is placed in an air bath and subjected to the temperature required (light oils should stand a test of 150° F. for 8 hours), and cooled and reweighed. The loss being figured in per cent. represents the amount evaporated. Gill says no oil should be passed that gives an evaporation of more than 4 per cent. with the above test. The United States Government requires that an oil should not lose more than 5 per cent. of its weight in 2 hours when exposed to 400° F.

RAILROAD SPECIFICATIONS AND STATE REQUIREMENTS

CHICAGO, MILWAUKEE, AND ST. PAUL RAILWAY COMPANY'S SPECIFICATIONS

41. Kerosene Oils.—These oils will be purchased subject to the following conditions and specifications: Dealers will be required to send to the purchasing agent a sample of each grade of oil they propose to furnish. Upon receipt of a carload of oil, one barrel will be selected at random and sampled. If this fails to pass all the following specifications of its class, the entire carload will be rejected and returned at the shipper's expense. If, after being accepted, any portion should be found damaged or inferior to the original sample, that carload will be rejected and returned as above. Packages must be strictly prime and in good order, with brand of oil and consignor's name stenciled on head with gauge marks, which will be subject to this company's verification. Oil containing glue or other foreign matter in suspension will be rejected. Oil that shows undue tendency to smoke or produce a white deposit on chimneys will be rejected.

42. Headlight oil, or so-called 150° fire-test oil, should have the following qualifications: (1) The oil must not flash below 110° F. when heated at the rate of 2° per minute, in closed-cup tester. (2) The gravity at 60° F. may vary between 46° and 50° Baumé. (3) The color must be clear, bright, and practically water white.

Winter oil must have a cold test not higher than 25° F. below zero, and is called *low cold-test oil.*

Spring and Fall oil must have a cold test not above zero, and is called *medium cold-test oil.*

43. Mineral seal oil, or so-called 300° fire-test oil, should have the following requirements: (1) The oil must not flash below 235° F. when heated at the rate of 2° per minute in closed-cup tester. (2) The gravity at 60° F. may vary between 39° and 42° Baumé. (3) The color must be clear, practically white, with little or no bloom.

NOTE.—The Elliott closed-cup tester (standard in New York, New Jersey, and Iowa) is used in determining the above flashing points.

BALTIMORE AND OHIO RAILWAY SPECIFICATIONS

44. All oil is purchased by weight. Prices should be given in cents per pound (6.57 pounds per gallon). Shipments of one or more barrels which are filled with oil, cloudy from the presence of glue, or which contain dirt, water, or other impurities, will be rejected.

45. **150° fire-test oil** (for office, switch, and station lamps) must have a flash test of at least 125° F. in a Tagliabue open cup, a fire test not below 150° F., and a cloud test not above 0° F. It must be water white in color and free from sulphur in any form; must be between 46° and 48° Baumé gravity at a temperature of 60° F.; must not flock when heated to a temperature of 270° F. for 1 hour, and must burn freely and steadily with the wick and burner used for this oil.

46. **300° fire-test oil** (for lamps in passenger cars) must have a flash test above 250° F., a fire test not below 300° F., and a cloud test not above 32° F. When heated to a temperature of 425° F. and held there for 5 minutes, the oil must remain clear and transparent, showing but a slight darkening and no separation of flocculent or other matter—either at this temperature or on cooling. It must be *standard white* in color and free from sulphur in any form; must be between 38° and 42° Baumé gravity at a temperature of 60° F.; must weigh 6.85 pounds per gallon and must burn freely and steadily with the wick and burner used for this oil.

47. **Signal Oil.**—This grade of oil shall be prime white in color, shall contain not less than 40 per cent., by weight, of prime lard oil, and shall show a flashing point not below

200° F. and a burning point not above 300° F. The test will be made in an open vessel by heating the oil not less than 15° per minute, and applying the test flame every 7°, beginning at 193° F. When heated to a temperature of 450° F. and held there for 5 minutes, the oil must remain clear and transparent, showing but a slight darkening and no separation of flocculent or other matter—either at this temperature or on cooling. The gravity may be from 31° to 34° Baumé.

UNITED STATES QUARTERMASTER'S SPECIFICATIONS

48. **Mineral oil** to be water white, of a flash point not lower than 135° F. nor higher than 150° F. The specific gravity to be not less than 48° Baumé.

49. The **packages** to contain the oil to be cans of capacity of 5 gallons each, to be made throughout of IX bright charcoal tin, strictly first quality. Each can to have on top, near one corner, a 1¼-inch closed screw cap of uncut zinc or a screw cap with nozzle as made by the Record Manufacturing Company, of Conneaut, Ashtabula County, Ohio; or with Marsh faucets (11 Broadway, New York), each kind on half the number of cans. Weight of cans to be not less than 3 pounds each. Cases to contain two cans and to be made of ⅞-inch pine throughout, close jointed (dressed on outside), and of such size as to admit of the cans fitting closely on the sides. A thin, loose piece of board, full width and height of can, to be inserted between them to fit sufficiently close to hold the cans tightly in place. A strip of pine of proper thickness to be inserted between the cans and top of case, to protect the screw cap. The cans and cases to be made in the best workmanlike manner. The cases to be strongly nailed with not smaller than 8-pennyweight nails. Great care must be used in casing the oil that the nails are not driven into the oil cans.

50. The **oil** will be inspected and tested at point of delivery by sworn inspectors employed by the United States, the instruments used being Tagliabue's pyrometer (closed cup) and Tagliabue's hydrometer.

51. The government inspectors heat the oil at 1° instead of 2° per minute in making these tests. Thus, the 135° F. requirement is about 128° F., as commonly taken on the Tagliabue closed tester.

COMPOUNDED OILS

52. **First-Grade Cylinder Oil.**—High-pressure cylinder oil for use in high-pressure cylinders: To be a mixture of the best petroleum stock and acidless animal oil, tallow oil being preferred. It must contain not less than 5 per cent. of pure, acidless animal oil, and have a flash point at not less than 550° F.; a fire test not below 600° F.; a cold test below 55° F.; and a gravity between 25½° and 27½° Baumé, at 60° F.

53. **Second-Grade Cylinder Oil.**—Low-pressure cylinder oil for use in low-pressure cylinders: To be a mixture of pure petroleum cylinder stock and acidless animal oil, tallow oil being preferred. It must contain not less than 10 per cent. of pure, acidless animal oil, and have a flash point of not less than 525° F.; a fire test not below 575° F.; a cold test below 40° F.; and a gravity between 26° and 28° Baumé at 60° F.

54. **Machine Oil.**—To be a free-running, hydrocarbon oil. May contain not more than 10 per cent. of acidless neatsfoot or tallow oil. The combined oil to be free from alkali or acid; flash point not less than 480° F.; gravity about 25° Baumé.

55. **Dynamo Oil.**—To be a free-running hydrocarbon oil. May contain not more than 5 per cent. of acidless neatsfoot or tallow oil. The combined oil to be free from alkali or acid; flash point not less than 402° F.; gravity about 28° Baumé.

56. The foregoing described oils must be free from all adulterations or mixture with animal, vegetable, or fish oils, grease, lard, or tallow, except as stated, and contain no tarry or suspended matter acid or alkali, glue or water.

METHODS OF TESTING THESE COMPOUNDED OILS

57. Flashing Point and Burning Tests.—Heat a small quantity of the oil in an open vessel, not less than 12° per minute, and apply the test flame every 10°, beginning at 250° F.

58. Precipitation Test for Tarry and Suspended Matter.—Mix 5 cubic centimeters of oil with 95 cubic centimeters of 88° gasoline, and if there is any precipitation in 10 minutes the oil will be rejected. This test is easiest made by putting 5 cubic centimeters of oil in a 100-cubic-centimeter graduate, then filling to the mark with gasoline and thoroughly shaking.

59. Test for Adulteration, Saponification, Etc.—This test depends on the property that mineral oils possess of not forming an emulsion when treated with alkali. Take 2 ounces of potassium hydrate, or of pure caustic soda, and dissolve in 8 ounces of distilled water. The sample of oil to be tested is put in a bottle, to which is then added at least an equal quantity of the potash or soda solution; shake well and allow to settle. Heating the mixture by plunging the bottle in a hot bath often assists this test. If the test is made in duplicate with a mineral oil of known purity, a comparison of the results obtained will be found valuable. Borax dissolved in water shaken into an equal quantity of mixed oil will be found to separate even small percentages of mineral oil. The borax solution must not be too strong, or a satisfactory result will not be obtained.

60. Test for Volatility.—When kept heated at 400° F. in an open vessel it must not lose more than 5 per cent. of its weight in 2 hours.

61. Test for Acid or Alkali.—It will be sufficient to wash a small quantity of the oil with distilled water; then drain off the water and test it with litmus paper.

TABLE V

STATE REQUIREMENTS

Alabama	No law.
Arizona	No law.
Arkansas	130° F. fire, Tagliabue open cup.
California	No law; cities, 110° F. flash.
Colorado	No law; cities, 110° F. flash.
Connecticut	110° F. fire, Tagliabue cup.
Delaware	110° F. fire, Tagliabue cup.
District of Columbia	120° F. flash.
Florida	130° F. fire, Tagliabue cup.
Georgia	120° F.
Idaho	No law.
Illinois	150° F. fire, Tagliabue.
Indiana	120° F. flash, Indiana cup.
Indian Territory	No law.
Iowa	105° F. flash, Elliott cup.
Kansas	110° F. fire, Tagliabue.
Kentucky	130° F fire.
Louisiana	125° F. flash, Tagliabue.
Maine	120° F. flash, Tagliabue.
Maryland	No law.
Massachusetts	100° F. flash, Tagliabue open cup.
Michigan	120° F. flash, Foster cup.
Minnesota	110° F. flash, Minnesota cup.
Mississippi	No law.
Missouri	150° F. fire, Tagliabue.
Montana	110° F. flash.
Nebraska	Over 100° F. flash, Foster cup.
Nevada	No law.
New Hampshire	100° F. flash, Tagliabue.
New Jersey	100° F. flash, 115° F. fire.
New Mexico	150° F. fire.
New York	110° F. fire, Tagliabue.
North Carolina	100° F. flash, Foster cup.
North Dakota	100° F. flash.
Ohio	120° F. flash, Foster cup.
Oregon	No law.
Pennsylvania	110° F. fire.
Rhode Island	110° F. fire.
South Carolina	No law.
South Dakota	110° F. flash, Foster.
Tennessee	120° F. flash, open cup.
Texas	No law.
Vermont	110° F. fire, Tagliabue.
Virginia	110° F. fire, Tagliabue.
Washington	No law.
West Virginia	No law.
Wisconsin	120° F. flash, Wisconsin cup.
Wyoming	No law.

REQUIREMENTS

62. Requirements for Bidders.—Bidders must state specifically in their respective proposals the name and kind of each oil, its flash test, and specific gravity. Proposals, to receive consideration, must be accompanied by a certified check in the sum of five hundred dollars ($500), payable to the order of the Secretary of the Treasury, as a guarantee of good faith. The check of the successful bidder will be retained, with the understanding that the whole, or so much thereof as may be required, shall be used by the Department to supply any deficiencies that may arise owing to the inferior quality of the oils delivered, or delays in filling orders. Proposals must also be accompanied by samples of oils in 6-ounce vials, which must be labeled with the bidder's name and description of the oil, properly packed, and addressed to the Storekeeper, Treasury Department, Washington, D. C.

63. State Requirements.—Table V gives the requirements for illuminating or burning oils for forty-eight States and Territories. Table VI gives the requirements for foreign countries.

TABLE VI

REQUIREMENTS OF FOREIGN COUNTRIES

Belgium	35° C., Granier closed cup.
Denmark	40° C., Danish cup, open.
Germany	21° C., Abel closed cup.
France	35° C., Granier closed cup.
Japan	46° C. or 115° F., open cup.
Austria	21° C., Abel closed cup.
England	23° C. or 73° F., Abel closed cup.
Holland	40° C., Parrish closed cup.

64. Table VII gives the weight per gallon of petroleum and its products for different specific gravities.

TABLE VII

PETROLEUM AND ITS PRODUCTS

Degrees Baumé	Specific Gravity	Pounds Per Gallon	Degrees Baumé	Specific Gravity	Pounds Per Gallon
10	1.0000	8.33	49	.7821	6.52
11	.9929	8.27	50	.7777	6.48
12	.9859	8.21	51	.7734	6.44
13	.9790	8.16	52	.7692	6.41
14	.9722	8.10	53	.7650	6.37
15	.9655	8.04	54	.7608	6.34
16	.9589	7.99	55	.7567	6.30
17	.9523	7.93	56	.7526	6.27
18	.9459	7.88	57	.7486	6.24
19	.9395	7.83	58	.7446	6.20
20	.9333	7.78	59	.7407	6.17
21	.9271	7.72	60	.7368	6.14
22	.9210	7.67	61	.7329	6.11
23	.9150	7.62	62	.7290	6.07
24	.9090	7.57	63	.7253	6.04
25	.9032	7.53	64	.7216	6.01
26	.8974	7.48	65	.7179	5.98
27	.8917	7.43	66	.7142	5.95
28	.8860	7.38	67	.7106	5.92
29	.8805	7.34	68	.7070	5.89
30	.8750	7.29	69	.7035	5.86
31	.8695	7.24	70	.7000	5.83
32	.8641	7.20	71	.6990	5.80
33	.8588	7.15	72	.6956	5.78
34	.8536	7.11	73	.6923	5.75
35	.8484	7.07	74	.6889	5.72
36	.8433	7.03	75	.6829	5.69
37	.8383	6.98	76	.6823	5.66
38	.8333	6.94	77	.6789	5.63
39	.8284	6.90	78	.6756	5.60
40	.8235	6.86	79	.6722	5.58
41	.8187	6.82	80	.6666	5.55
42	.8139	6.78	81	.6656	5.52
43	.8092	6.74	82	.6619	5.50
44	.8045	6.70	83	.6583	5.48
45	.8000	6.66	84	.6547	5.45
46	.7954	6.63	85	.6511	5.42
47	.7909	6.59	90	.6363	5.30
48	.7865	6.55	95	.6222	5.18

MANUFACTURE OF GAS

(PART 1)

INTRODUCTORY

HISTORY

1. Early Experiments.—Probably the most important industry involving the destructive distillation of coal is the manufacture of illuminating gas. Its discovery dates back as far as 1727, when Clayton, Bishop of Cork, and Doctor Hales observed the escape of gas on heating coal, and in 1767 Doctor Watson observed that combustible gas could be conveyed through pipes. Professor Pickel lighted his laboratory in Würzburg, Germany, in 1776, with gas obtained from the destructive distillation of bones. About the same time Dundonald experimented with coal gas in England. His object was the production of gas tar as a by-product of coke manufacture. The workmen fixed iron pipes in the condenser in which the tar was deposited, and kindled the escaping gas for light in the night. Strictly speaking, the beginning of gas lighting took place in 1792, when Murdoch lighted his house and works at Redruth, England, with gas obtained from coal. His process did not become known on the European continent until about 10 years later. The first gas illumination on a large scale was installed by Murdoch in 1802, at the works of Watt and

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Boulton, the Soho Foundry, near Birmingham. In 1804 he fitted up a similar installation at a cotton-spinning establishment in Manchester. From this time the use of gas became extended, but was mostly confined to factories. Its introduction into daily life dates from 1812, when the streets of London were lighted with gas. In 1824 gas lighting was introduced into Germany.

2. Destructive Distillation.—As has been previously mentioned, gas is the product of the destructive distillation of coal, etc. The term **destructive distillation** is applied to the process of heating a complex substance to its point of decomposition. This is generally accomplished by performing the operation in a closed retort, as in coal-gas manufacture; but it may also be effected by the limited combustion of the substance to be distilled, so that the heat generated destructively distils the remainder, as, for instance, in charcoal burning and the manufacture of coke. The class of substances usually treated are such carbon compounds as are split up at a high temperature with the evolution of gaseous as well as liquid products, a residue relatively richer in carbon being left—a change that is indicated by the term *carbonization*. In the first class are coal and shale, and in the second, wood, bones, etc.

3. Cumulative Resolution.—It will be understood that the essential difference between the process of destructive distillation and that of ordinary distillation (such as that of alcohol, for instance) consists in the fact that the substances collected in the receiver in the former operation do not proexist in the retort; they are products of the process and not educts. The nature of the products obtained is dependent on the temperature, and, in a minor degree, on the pressure. It has been found that at high temperatures aromatic hydrocarbons—that is, those of the benzene and homologous series—predominate in the liquid products, while at low temperatures hydrocarbons of the fatty, or paraffin, series are present in greater amount. To the kind of change caused by destructive distillation, the term **cumulative resolution** is sometimes applied, by which is meant the polymerization of the substance distilled in conjunction with the removal of water from it. Thus, cellulose, $n(C_6H_{10}O_5)$, the typical constituent of wood, may be viewed as suffering decomposition by the following steps:

$$\begin{aligned} C_6H_{10}O_5 - H_2O &= C_6H_8O_4 \\ C_6H_8O_4 - H_2O &= C_6H_6O_3 \\ C_6H_6O_3 - H_2O &= C_6H_4O_2 \\ C_6H_4O_2 - H_2O &= C_6H_2O \\ C_6H_2O - H_2O &= C_6 \end{aligned}$$

4. It is further assumed that the organic matter in coal and shale contains carbon, which may be represented as existing in groups of C_2, so that its ultimate composition, as far as the carbon is concerned, is expressed by nC_2. This group can be traced in the products of destructive distillation. It must be noted, however, that it has been shown by Ramsay that pure cellulose yields acetic acid and methyl alcohol among its products of distillation, and that their formation can scarcely be explained by the example of cumulative resolution just given. The whole question in its theoretical bearings is complicated by the fact that the substances that it is customary to distil destructively are mixtures, and that numerous independent reactions probably take place. Thus, in the destructive distillation of wood, an exothermic change occurs at one point of the reaction, while with pure cellulose no such effect has been observed. A change of this kind may be attributed to the oxidation of a portion of the hydrogen or carbon of the material distilled, at the expense of another portion richer in oxygen, the reaction being induced by the attainment of a particular temperament. Whatever may be the precise mechanism of the changes involved in destructive distillation, the products usually include hydrogen, water, carbon monoxide, and carbon dioxide; gaseous, liquid, and solid hydrocarbons of several series and their oxygen derivatives;

ammonia, organic bases, and cyanides (if the substance contained nitrogen); and hydrogen sulphide, carbon disulphide, and sulphocyanides.

As coal gas was the first gas manufactured, we shall consider its manufacture first, following with water gas, acetylene, etc.

MANUFACTURE OF COAL GAS

RAW MATERIAL

5. Bituminous coals are distinguished from anthracite coals by their property of softening and apparently fusing when subjected to a temperature below that at which combustion takes place. This fusion indicates the commencement of destructive distillation, when solid, liquid, and gaseous compounds are formed. Bituminous coal is essentially a coking coal, and as such is, to a very great extent, employed in the coking regions of Western Pennsylvania. It is black or grayish black, possesses, as a rule, a resinous luster, and is rather friable, breaking up into cubical fragments. On ignition it burns with a decidedly yellow flame. When it is heated to bright redness, without access of air, as in retorts and ovens, volatile hydrocarbons, water, etc. pass off. Coal having a large percentage of hydrogen will yield more volatile substances at the temperature of distillation and less carbonaceous residue than others that may contain less hydrogen and more carbon, that is, approaching anthracite in composition.

6. Coking and non-coking coals are very similar as far as their chemical composition is concerned; coking coal, however, contains less volatile matter. Non-coking coal does not possess the property of fusing to a compact mass; it retains its original form, and the coke thus obtained has no commercial value.

7. Cannel coal is much more compact than either gas or coking coal. It has a dull grayish-black or dark-brown color, and burns readily with a clean flame. It is comparatively hard and does not soil the hands. It is found in West Virginia and in many of the Western States. Destructively distilled, it yields a larger amount of volatile matter and ash, with much less coke, than the bituminous coals.

8. The following tables, taken from the Reports of the Geological Survey of Pennsylvania, show the analyses of some of the more important varieties of American gas coals, coking coals, and non-coking coals.

TABLE I

GAS COALS

Constituents	Westmoreland Coal Company			Pennsylvania Gas Coal Company		
	South Side Mine	Foster Mine	Larrimer, No. 2	Irwin, No. 1	Irwin, No. 2	Sewickley
Water at 225°..	1.410	1.310	1.560	1.780	1.280	1.490
Volatile matter	37.655	37.100	39.185	35.360	38.105	37.153
Fixed carbon..	54.439	55.004	54.352	59.290	54.383	58.193
Sulphur.......	.636	.636	.643	.680	.792	.658
Ash...........	5.860	5.950	4.260	2.890	5.440	2.506
Total........	100.000	100.000	100.000	100.000	100.000	100.000

TABLE II

COKING COALS

Constituents	Connellsville, Frick & Co.	Bennington, Cambria Iron Company	Broad Top, Barnet	Broad Top, Kelley	Cumberland	Huntingdon County, Alloway Colliery.
Moisture......	1.260	1.400			1.10	.250
Volatile matter	30.107	27.225	16.00	19.68	15.30	14.510
Fixed carbon..	59.616	61.843	74.65	71.12	73.28	77.042
Sulphur.......	.784	2.602	1.85	1.70	1.23	1.338
Ash...........	8.233	6.930	7.50	7.50	9.08	6.860
Total........	100.000	100.000	100.00	100.00	100.00	100.000

TABLE III

NON-COKING COALS

Constituents	Mercer County, Pa. Sharon Coal	Youngstown, Ohio	Mercer County, Pa.	Straitsville, Ohio	Brazil, Ind.
Moisture	3.790	3.60	3.80		
Volatile matter	35.300	32.58	25.49	36.50	40.15
Fixed carbon	53.875	62.66	68.03	55.60	57.20
Sulphur	.675	.85	1.04	.96	.75
Ash	6.360	1.16	1.70	6.94	1.90
Total	100.000	100.00	100.06	100.00	100.00

FINISHED PRODUCT

9. Composition of Coal Gas.—As we have already seen, coal gas is a mixture of various organic and inorganic compounds, the amounts of which in a given volume of gas vary somewhat according to the kind of coal used and the temperature at which it is carbonized. The following may be considered the average composition of purified coal gas, from which the carbon dioxide has not been removed.

Hydrocarbon vapors	.6
Heavy hydrocarbons	4.4
Carbon dioxide	3.4
Carbon monoxide	10.1
Marsh gas	30.6
Oxygen	.3
Hydrogen	45.9
Nitrogen	4.7
Total	100.0

In the unpurified state, coal gas also contains ammonia, hydrogen sulphide, and a small percentage of other compounds, among which carbon disulphide predominates. Of the impurities, ammonia and hydrogen sulphide are carefully removed, but no extra efforts are made to remove the small amount of carbon disulphide that might escape the washer.

10. When the gas is driven off by the destructive distillation of the coal, it contains a certain proportion of hydrocarbons that volatilize only at a high heat, and these begin to condense as soon as the gas begins to cool, forming *tar*. The non-volatile part of the coal, consisting of the so-called fixed carbon and inorganic matter constituting the ash, is called *coke*. In addition to this, certain of the hydrocarbons attach themselves to the hot walls of the retorts in which the destructive distillation takes place, forming what is known as *retort carbon*. This retort carbon interferes with the heat-radiating power of the retort and must be removed at intervals, these intervals varying from 2 weeks to 3 months, according to the conditions under which the retorts are worked.

The length of time required to entirely drive off the gas from the coal varies according to the heat to which it is subjected, less time being required with a high than a low temperature. It is now almost the universal custom to have the retorts at a sufficiently high temperature to drive off the gas in about 4 hours. Under this condition, a long ton (2,240 pounds) of good gas coal should produce about 10,000 cubic feet of gas of 17 candlepower, about 1,400 pounds of coke, nearly 12 gallons of tar, and 4 pounds of ammonia. With coals of inferior quality, these figures, of course, vary to a certain extent.

11. By-Products.—Coke, tar, and **ammonia** are the by-products of the coal-gas manufacture. As a rule, gas coke is not suitable for blast-furnace work, as it does not possess the necessary hardness to support the heavy charges, but it makes an excellent domestic fuel. It burns freely with a light draft, it is practically smokeless, and is clean to handle. Most gasworks find a ready market for all that is not used about the works. Coke is sold either by the weight or, especially in smaller quantities, by measure. A cubic foot of gas coke weighs from 23 to 32 pounds, and a heaped bushel from 35 to 42 pounds; the average weight of a bushel is usually considered 38 pounds.

In the early history of gas manufacture there was very little demand for the tar, and it was frequently allowed to run to waste or was burned under the benches or at the boilers in the works. The progress of organic chemistry has developed large industries that use coal tar as a basis of their manufacture. More than 600 products, among which are the endless variety of aniline dyes, organic acids, such as carbolic and picric acids, etc., are now made from it. Tar is usually sold at so much per gallon.

Ammonia is produced during the destructive distillation of coal by the combination of hydrogen and nitrogen evolved. It is readily absorbed by water; at ordinary temperatures 1 volume of water absorbs 600 volumes of ammonia, but an increase in temperature decreases this absorption power. In gasworks the ammonia is removed from the gas by allowing it to come in contact with water, the supply of which is so regulated that a weak ammoniacal solution is produced, which is known as **gas liquor.**

PROCESS OF TREATMENT

12. Distillation of Coal.—The distillation of coal is carried on in special retorts. Those used in most American works are, in cross-section, shaped like the letter **D**, the flat side of the **D** being turned down and serving as the bottom of the retort. They are about 15 inches high inside, about 26 inches wide, and from 9 to 10 feet long if single-ended, and from 18 to 20 feet long if double-ended. In most works of moderate size, single-ended retorts are used. These retorts, formerly made of cast iron, were rapidly destroyed, the destruction being caused by the heat of combustion of the fuel used, by the sulphur in the coal gas forming sulphide of iron, and by the carbon, which, as a carbide of iron, graphitic in appearance, forms layers within the retort from 1 to 2 inches thick; the oxygen of the air has also a deleterious influence, especially on retorts heated to redness. They are now made of fireclay, with walls about 4 inches thick. Retorts are usually set horizontally, either singly or in groups of any number up to nine. A group of two or more retorts, heated by a single furnace, is called a **bench.** In this country, benches of threes are mostly used in small works, and benches of sixes in works of moderate size. Very large works, where machinery can be used to handle the coke and coal, prefer benches of nines. When a number of benches are built up side by side, such an aggregation is called a **stack.** Fig. 1 represents a bench of sixes.

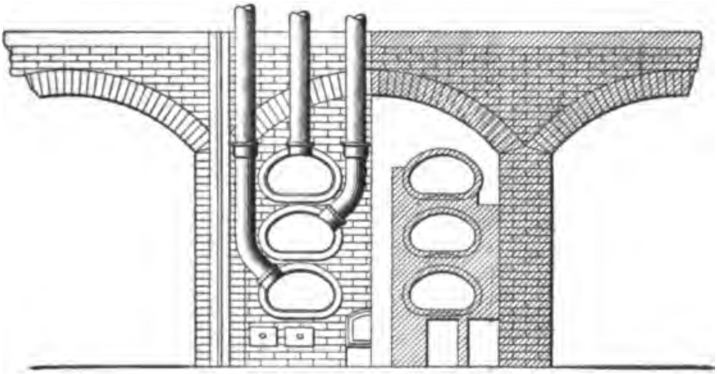

FIG. 1

13. Furnaces.—The furnace is built into a bench below the retorts, or it is placed just in front of and below the main part of the bench, depending on the kind of furnace used. There are two general styles of furnaces, namely, the **straight-fire furnace** and the **regenerative furnace.** The straight- or direct-fire furnace is set with the grate bars as near the level of the lower retorts as possible, and a comparatively shallow fire is maintained, the direct heat from the fire being relied on to bring the retorts to the proper temperature. The regenerative furnace is placed much lower in the bench and a deep fuel bed is kept, to which the supply of air is so regulated that only an incomplete combustion takes place, thus producing as large an amount of carbon monoxide as possible. Air passages, known as secondary air flues, enter the bench on each side of the furnace and pass between two flues, through which the furnace connects with the chimney at the back of the bench. The secondary air flues communicate with the furnace by a number of holes along each side of the furnace wall, just above the level of the top of the fuel, so that after the air that enters this way has been heated by the hot flues, it meets the carbon monoxide that comes up from the fuel bed. Combustion below and around the retorts then takes place in a very similar manner to that seen in a Bunsen burner, and an even, intense heat is produced. The regenerative furnace is more expensive, as far as the original cost is concerned, than the straight-fire furnace. Its advantage is in the saving of fuel; with a regenerative furnace, less than 30 per cent. of the coke produced is required, while with the straight-fire furnace 40 per cent. is required.

14. A pan containing water is always placed below the grate bars of a furnace, and the steam that rises from this keeps the bars cool and prevents largely the formation of clinkers. It is of advantage to have, in addition to this pan, a small steam pipe run into the furnace below the grate bars, so that a little steam may be admitted all the time, and also that a considerable quantity may be admitted for a couple of hours just before the fire is cleaned.

The fires should be cleaned about once in 24 hours in a regenerative furnace, and more frequently in a straight-fire furnace. Prior to the cleaning, the fire should be allowed to burn low, and it is then either pushed back on to the sloping bridge wall at the rear of the furnace, or else false grate bars are driven in about 1 foot above the regular bars. The ashes and clinkers are then removed as quickly as possible, the false bars, if used, are removed, and the fuel pulled forwards on to the grate bars. The furnace should then be charged again with fresh coke, preferably hot, just as it is drawn from the retort.

It is always better to use hot coke as furnace fuel, and a movable chute can be used, so that the coke, as it is drawn from the retorts, slides down into the furnace charging door, which is placed just below the lower retorts.

15. Dampers.—Usually each furnace connects with its own chimney or stack that rises at the rear of the bench, and dampers are always provided at the stack as well as at the main air-inlet below the grate bars and at the inlet of the secondary air flues. To maintain a good fuel economy, a bench should be damped as much as possible at the stack damper, allowing only just sufficient draft on the fire to keep up the required temperature.

In regenerative furnaces, the dampers on the main and secondary air supply must be so regulated that a proper combustion takes place above the fuel bed, as has already been mentioned. To obtain this condition is a matter of experiment and experience, and no hard-and-fast rules can be laid down. It may, however, be stated that if flames can be observed in the outlet flue at the stack damper, not enough secondary air is admitted, and the secondary air supply should be increased, little by little, until these flames do not appear except just after fresh fuel has been put in the furnace. If the secondary air flues appear to be getting continually cooler, too much secondary air is admitted and the supply should be decreased.

In all heat regulations it should be remembered that the temperature of a bench changes comparatively slowly. Only one set of dampers should be moved at a time, and at least several hours should be allowed to pass under the new, or rather corrected, conditions, in order that the effect of the change may be fully realized.

16. Mouthpieces.—The retorts are set in above the furnace, with the closed end against the rear wall of the bench and the open end flush with the front. The upper retorts are supported on the lower ones by blocks of firebricks, so that the heat has free access to almost all parts of the external surface of the retorts. On the open end of the retort an iron mouthpiece is bolted. This mouthpiece, shown in Fig. 2, is provided with a door, or lid, *a* and a socket *b* for the pipe known as the stand pipe, through which the gas leaves the retort. The lid, which must be removed each time the retort is charged, is clamped with a cotter bar and screw *c* and luted with clay to make it gas-tight.

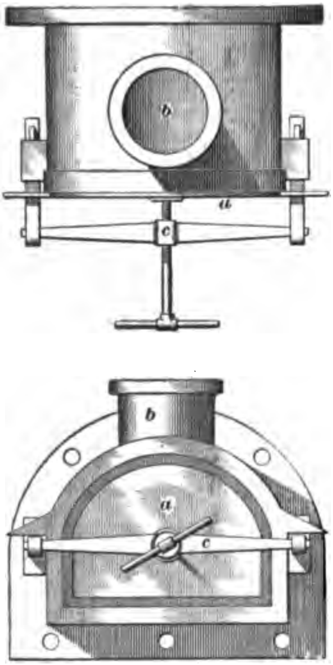

FIG. 2

In most modern works, however, this mouthpiece has been improved, as shown in Fig. 3; the lid *a* has a ground face, which avoids luting, and is held in place by a swinging cotter bar *b*, which is provided with an eccentric tightening lever.

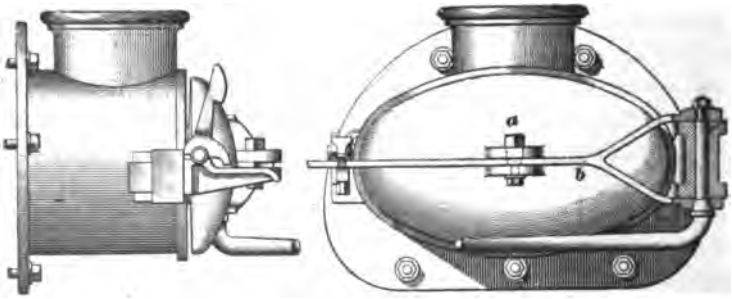

FIG. 3

17. Arrangement of Pipes.—The stand pipes run up from the mouthpieces to above the bench, where they connect by the bridge pipes with the dip pipes that run down into the hydraulic main. The arrangement of the pipes is shown in Fig. 4, where *e* represents the mouthpiece shown in detail in Fig. 3, *d* the stand pipe, *b* the bridge pipe, *c* the dip pipe, and *a* a section of the hydraulic main. Removable caps are always provided at the top of the stand pipe and dip pipe, so that they may be easily cleaned. In the most modern construction, the top of the stand pipe, bridge pipe, and dip pipe are all united in one piece that may be readily removed. The hydraulic main is a long pipe, either of cast or wrought iron, that runs along on top of the bench at the front. It should not be set directly on the masonry, but should be separated from it by a space of several inches, as shown in Fig. 4. Through the top of the hydraulic main the dip pipe *c* descends a little over half way to the bottom. It is essential that the bottoms of all the dip pipes should be set exactly at the same level, so that, when the hydraulic main is partly filled with water or tar, all the dip pipes entering it will be sealed off at the same time.

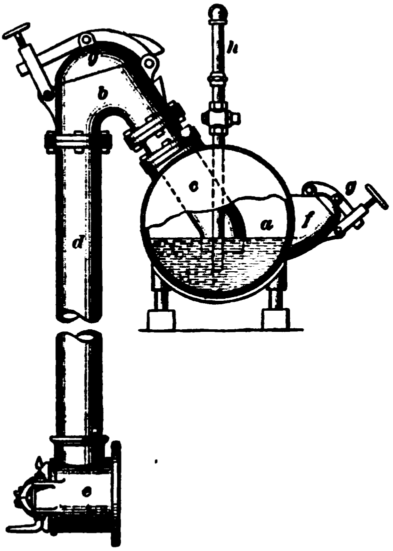

FIG. 4

18. Maintaining the Seal.—In the early days of gas making the tar that accumulates in the hydraulic main was allowed to form the seal, but it was soon discovered that a considerable proportion of the heavy hydrocarbons was absorbed by the tar and the illuminating value of the gas reduced, so that it is now the practice to keep the level of the tar several inches below the bottoms of the dip pipes, and to allow enough weak ammoniacal liquor to remain in the main to seal off the dip pipes. By allowing the tar to flow out through an inverted siphon, the outer end of which is set at a level above that desired for the tar in the main, the weight of the ammonia water on top of the tar in the main will drive the latter out until such a level is reached that the pressure maintained by the tar plus the water in the hydraulic main is equal to that maintained by the column of tar in the outer leg of the siphon. The equilibrium thus established will remain constant as long as the level of the ammonia water is kept the same, and this is insured by an overflow, so that a tar overflow and an ammoniacal-liquor overflow are thus both maintained at the same time, and the respective levels of the tar and ammoniacal liquor in the hydraulic main are kept always constant.

19. The object of maintaining the seal in the hydraulic main is to prevent the gas from other retorts coming back through the stand pipes and out of the mouthpieces of any retort that may be opened for charging. The gas as it is liberated in the retort easily bubbles through the slight seal of the dip pipe, and reaches the space in the hydraulic main above the water; but it cannot be forced out through a dip pipe except by a very high pressure, because the area of the water that is inside the dip pipe is only a small fraction of the area of the surface of the water in the entire hydraulic main; and consequently, if the pressure in the hydraulic main is sufficiently increased to drive down the level of the water there even a fraction of an inch, the water in the dip pipe would rise up several inches and form an effective seal.

In order to keep the tar, which sometimes becomes quite stiff, flowing towards the overflow that is usually set at the end of the hydraulic main, which may be extended to connect with several benches, the hydraulic main is sometimes made so that its bottom slopes in the direction of the overflow; and in other cases jets are introduced into the tar, pointing towards the overflow, and ammoniacal liquor is pumped through these, so that the tar is kept moving towards the overflow. At the back of the hydraulic main, cleaning doors are always set at frequent intervals, so that the heavy tar and pitch that is sure to accumulate may be removed. There is a diaphragm set inside these doors reaching down below the water level, so that, while tools may be inserted for cleaning the bottom of the main, the gas cannot escape.

The hydraulic main is always provided with an outlet pipe for the gas, which is generally known as the foul main.

20. Firing Retorts.—After a bench has been built, it is advisable to allow it to stand idle for some time, so that the masonry may have a chance to dry out. During this drying period, all the dampers, as well as the numerous plug holes in front of the bench, which are provided for cleaning the flues, should be left open to allow free access of air. When it becomes necessary to fire up the bench, the plugs should be put in, and the secondary air dampers, if there are any, should be closed. The ash-pan should be filled with water, and a small fire maintained in the furnace. The fire should be kept very low for several days while the bench is warming up, and then should be increased only very gradually until the insides of the retorts are seen to be heated to a dull red when the lid is removed. The secondary dampers should then be opened slightly and the bench brought to a working heat, which means that the retorts must show a bright red color inside. The stand pipes, which should have been fastened into the sockets on the mouthpiece by wooden wedges, should then be made up either with lime or a mixture of iron borings and fireclay (in the proportion of 1 : 4), the mixture being moistened by either a solution of ammonium chloride and water or by weak gas liquor. The retorts should never be charged until they have assumed a good red heat, and the charge should be light, say about 150 pounds of coal per retort. A slow and careful firing will greatly increase the life of a retort. Good retorts should last at least 2 years, but with careful firing and handling they will even last for 3 years.

When it becomes necessary to renew all the retorts, the entire front wall of the bench below the arch is torn out, the retorts replaced, and the front wall rebuilt. Should any one retort become badly damaged while the rest of the retorts in the same bench are still good, it may be bricked up at the mouth and left in that condition until such time as the others need repairs.

21. When it is necessary to shut down a working bench, the retorts should be charged nearly full of coal and the stack dampers should be nearly closed. All other dampers should be tightly closed, and all cracks and holes should be carefully luted up with clay. After several hours, the stand pipes should be opened at the top, to prevent any chance of gas backing into the retorts from the hydraulic main and forming an explosive mixture. Neither the bench nor the retorts should be disturbed for several days, until the former is quite cool, when the latter are opened and the coke withdrawn.

22. Where regenerative furnaces are used, the retort house is frequently built with a cellar, in which the cleaning doors of the furnace are located. Free access is thus allowed to the fires, and the furnaces are cleaned more easily and rapidly. A fireproof floor, preferably of brick, supported by iron beams, is placed over the cellar, through which chutes for the coke descend, so that the coke, as it comes from the retorts, falls into iron wagons or barrows. Where there is no cellar, as is mostly the case where straight-fire furnaces are used, a pit covered by an iron plate is placed in front of each bench, the cleaning door opening into it.

The coal is usually brought on to the retort-house floor in iron wagons, holding enough coal to charge three retorts. These wagons are provided with a hinged side, so that it may be lowered and the coal shoveled conveniently from the wagon into the retort.

23. Clogging of Stand Pipes.—One of the greatest difficulties met in coal-gas manufacture is to prevent successfully the clogging of the stand pipes. Stand pipes are clogged by the accumulation of either pitch or lampblack, or both. This clogging of the stand pipes is either due to too high or irregular heat, or, what amounts to practically the same, irregularity in charging. Where high heats are habitually carried and as much gas extracted per ton of coal as possible, the accumulation of pitch and lampblack is inevitable, and the only remedy is constant care to keep the pipes clean. In any case, an instrument called an auger, shown in Fig. 5, should be run into the lower end of the stand pipes every time the retorts are opened, and it is good practice to remove the cap at the top of the stand pipe and run a long auger, almost as large as the inside diameter of the stand pipe, through each pipe at least once every 12 hours. This should be done as soon as the retort is opened, and before the coke is drawn.

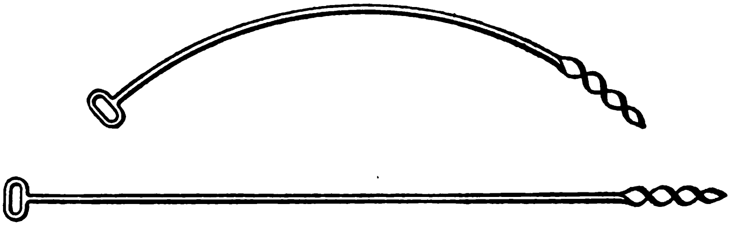

FIG. 5

In case the auger will not go through the stand pipe, a little wood should be placed in the mouthpiece, and the inside of the stand pipe burned out until the auger will go through. As soon as any stand pipe shows signs of clogging, by the gas leaking around the mouthpiece, the retort should at once be opened and the stand pipe burned out. If the stand pipe is entirely clogged up, an iron pipe or bar must be used to poke from the top and open a hole through it. In extremely obstinate cases it is advisable to heat the end of the pipe or bar. Where the stand pipe is clogged with soft pitch, it frequently happens that the hole will close up as soon as the bar is withdrawn. In this case the hot bar must be used until a permanent hole is established. A wood fire should then be built in the mouthpiece, so that the flame will draw up through the open stand pipe. As soon as the pitch is sufficiently heated, it will burn inside of the standpipe, leaving a hard, scaly residue that can be readily dislocated by means of an auger.

24. An apparatus that will greatly facilitate this work, by providing a strong draft up the stand pipe, is shown in Fig. 6, and may be made as follows: Take a piece of ½-inch pipe *a* about 2 feet long; place a ½″ × ⅜″ elbow *b* on one end of it, and screw a piece of ⅜-inch pipe about 3 feet long in the elbow. On the end of this pipe place a return bend, or two elbows in the form of a return bend *d*, and in the other end of the return bend place a nipple *e*, about 3 inches long and reduced so that the outer end has an inside diameter of about ⅛ inch. The ½-inch pipe *a* should be connected with a high-pressure steam line, and the nipple end *e* dropped into the stand pipe and the steam turned on. As a powerful draft is thus created up the stand pipe, it will be burned out in much shorter time than is otherwise possible.

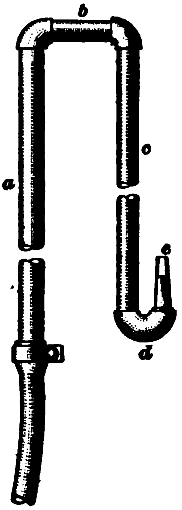

FIG. 6

25. In case the clogging in the stand pipe is so hard that no hole can be made through it, the stand pipe must be taken down and a fire built around it until it is red hot and the clogging burned out. When a stand pipe is opened for cleaning, it should be accomplished as quickly as possible, and on no account should the stand pipe be allowed to cool. It must be remembered that the cooler the stand pipe gets, the more difficult it is to clean it efficiently

The dip pipes should also be cleaned at regular intervals, as they are just as liable to be clogged up as the stand pipes.

26. Opening and Cleaning the Retort.—It should be remembered that a retort should not be opened quickly, owing to the accumulation of gas. If it becomes necessary to open the retort while in operation, the lid should be slacked off a little and the escaping gas ignited. As soon as this is done, the lid may be safely thrown wide open. Neglect of this precaution is sure to produce a violent explosion and injury to the men around the bench.

27. The substance called carbon, accumulating on the inside of the retort, is deposited with decreasing rapidity as the pressure in the retort is reduced. In older works, where no exhauster is used, and where the pressure of the holder and the back pressure of all the other apparatus is on the retorts, it is sometimes necessary to remove the carbon from each retort once a week. In works where the exhauster is run so that the hydraulic main is about at atmospheric pressure and a light seal is carried on the dip pipes, the carbon need be removed from the retorts only every 2 or 3 months. Where steam is not readily available, the following method to remove the carbon may be employed: An iron pipe from 3 to 6 inches in diameter is introduced into the retort, so that it reaches nearly to the back, and the mouthpiece is bricked up with brick and fireclay, care being taken not to stop up the opening of the stand pipe. The cap at the top of the stand pipe, is removed, and a draft at once starts that draws air in through the pipe to the back of the retort. As the hot air leaves the pipe at the rear of the retort and starts forward to escape through the stand pipe, combustion of the hot carbon on the sides of the retort begins, and it becomes detached from the sides of the retort. By continuing this for some time, all the carbon may be removed and consumed.

28. Charging the Bench.—The bench being at a good working heat, the proper amount of coal is shoveled into it.

This varies from 200 to 400 pounds per retort, according to the size. It is essential that the coal should be laid in an even layer from the rear to the front, and not piled up in any one place, as in that case part of it will be heated too hot while the gas will not be all driven out of the other part.

Considerable skill is necessary to charge the retorts properly, and a peculiar shovel, shown in Fig. 7, which is not bent as much as ordinary shovels are, is used.

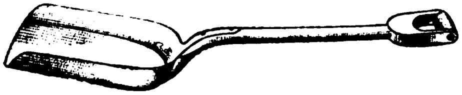

FIG. 7

In some cases, long scoops that will fill the retort in one or two charges are used. Such a scoop, shown in Fig. 8, is operated by three men: Two of them lift the front end of the loaded scoop on a bar *b*, and the third lifts the rear end. The scoop is then run into the retort, dumped, withdrawn, and the operation repeated.

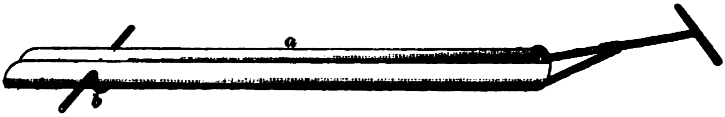

FIG. 8

The charge is allowed to remain in the retort for 4 hours, at the end of which time, if the bench is at the proper temperature, the gas is about all driven off. The retort is then opened and the coke pulled out by a rake, or draw bar, shown in Fig. 9. A part of the coke is usually charged into the furnace, preferably hot, just as it comes from the retort, and the rest is quenched with water and wheeled out on the coke pile.

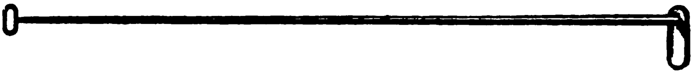

FIG. 9

29. The hot gas passes up the stand pipe and across the bridge pipe, down through the dip pipes, and bubbles out through the seal into the hydraulic main. Here it cools somewhat, and some of the less volatile parts are condensed into tar and ammoniacal liquor. The gas in the hydraulic main is at a high temperature, and contains as chief impurities tar, ammonia, and hydrogen sulphide, all of which must be removed, besides carbon dioxide, which may or may not be removed.

A hot tar scrubber, which is a steel shell full of baffle plates, is frequently placed between the hydraulic main and the first condenser, in works where no other form of tar extractor is used.

From the hydraulic main the foul main runs to the exhauster. There is often a condenser on this line, so that the gas is cooled before it gets to the exhauster. This condenser is usually a tall steel cylinder, through which numerous tubes are run, so that cool air entering at the bottom passes up through the condenser. The amount of cool air so introduced may be regulated by a damper at the top. The inlet of the condenser is at the top, and the outlet at the bottom, and the gas passes down it around the tubes and is so cooled somewhat. An air condenser of this type is shown in Fig. 10.

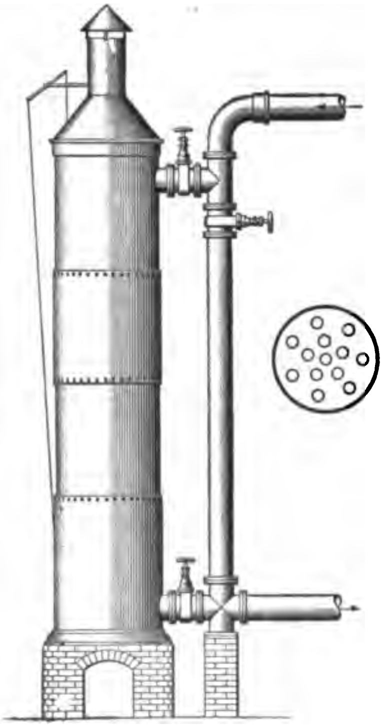

FIG. 10

30. **The Exhauster.**—This machine consists of a rotary pump, driven by a steam engine, which takes the gas away as fast as it is made. The arrangement is shown in Fig. 11. The throttle valve on the steam engine is connected by a lever to a tank shaped like an inverted cup and floating in water. A pipe connects this tank with the hydraulic main, so that, when the volume of gas increases in the main and more pressure is produced, the steam valve is opened and the speed of the engine increased. In this way, the surplus of gas is speedily removed and the pressure brought back to the desired point. By means of balance weights, any desired pressure may thus be maintained in the hydraulic main. As has been previously stated, it is usually kept at about atmospheric pressure.

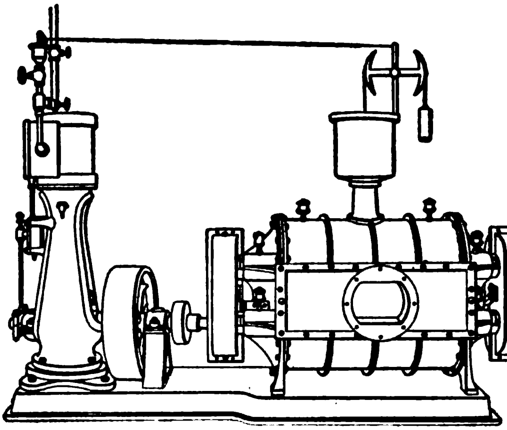

FIG. 11

31. Tar Extractor.—In many works the gas passes on from the exhauster, under pressure, to the **tar extractor.** The Pelouze and Andonin type, shown in Fig. 12, is most frequently used. It is arranged with two or more sets of perforated plates, set so that the holes in the plates do not come directly opposite each other. The vesicles, or bubbles, of gas and tar are broken up by friction and the tar is driven down. The plates dip into a tar seal at the lower

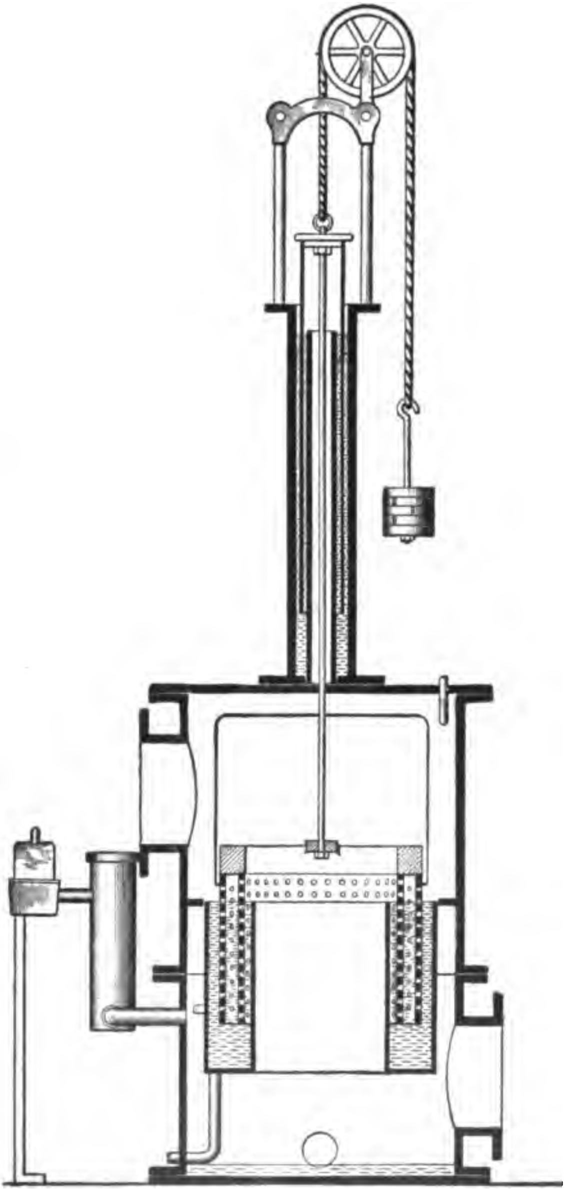

FIG. 12

end, and a counterbalance weight and drum are so arranged that as the volume of the gas passed increases or diminishes, the plates are automatically raised or lowered and more or less slots uncovered, so that the pressure remains practically constant. The tar from the extractor, together with that from the hydraulic main, is run off to the tar well.

32. Condensers.—From the extractor, the gas, which should now be between 110° and 130° F., passes to another condenser. This is generally the **air-cooled** type, as shown in Fig. 10. Finally the gas reaches the third or last condenser, known as the **multitubular water condenser.** This is very similar to the one shown in Fig. 10, except that the space surrounding the gas pipes is kept full of cold water. Its chief advantage is that the temperature in it can be readily controlled by regulating the water supply. After the gas has been through the last condenser, its temperature should be below 70° F.

Another condenser, extensively used in England, is shown in Fig. 13 and is known as an **atmospheric condenser.**

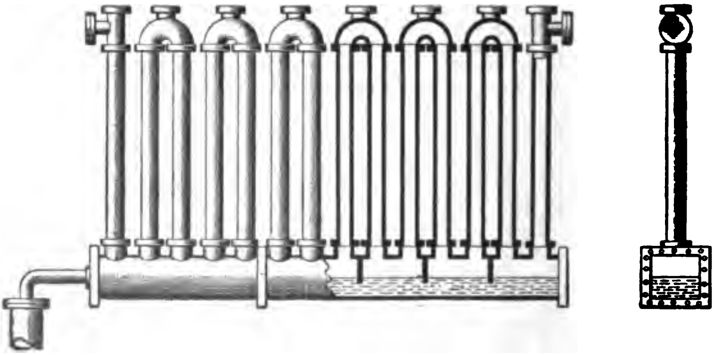

FIG. 13

It consists of a number of pipes, through which the gas is made to pass in succession. The radiation from the surface of the pipes allows the heat of the gas to escape, while the continued friction of the gas against the inside of the pipes breaks up any tar vesicles that may be present.

33. Washers.—After having passed through the condenser, the gas is ready for the **washer,** where the bulk of the ammonia, part of the hydrogen sulphide, other sulphur compounds, and carbon dioxide are removed. A very simple but efficient apparatus for the work is a wash box of the type shown in Fig. 14. In this washer the gas enters above the surface of the gas liquor, in several long troughs, from which it passes down and bubbles up through the liquor. This agitation converts the surface of the gas liquor into foam and spray, so that the gas is brought into thorough contact with the liquor, whereby ammonia, etc., is absorbed. By regulating the rate at which the gas liquor flows through the washer, the overflow may be made weaker or stronger as desired.

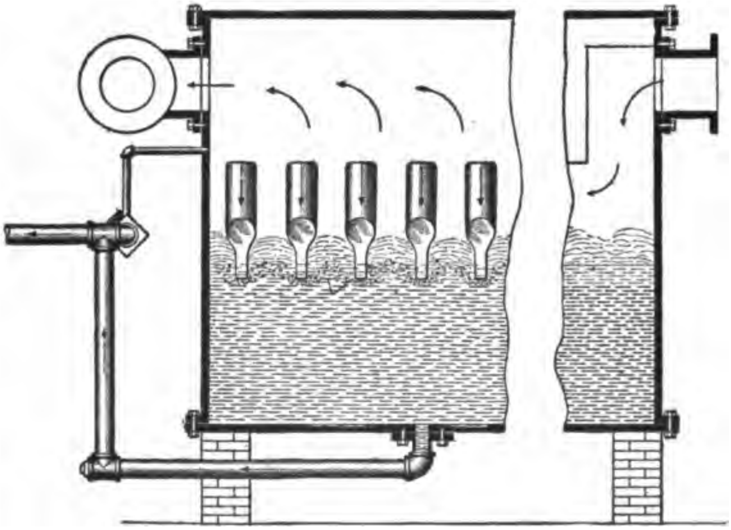

FIG. 14

Another form of washer, known as the **standard washer,** is shown in Fig. 15. It consists of a long iron cylinder *a*, through which an iron shaft *b* runs, which is turned by a belt and pulley *c*. The cylinder throughout is divided into smaller compartments, as seen in the illustration. The compartments are made of sheet metal, wood, or slate, and are attached to the revolving shaft *b*. They are filled with brushes. The gas enters at *d* and leaves, after passing through all the compartments. The gas-outlet pipe serves at the same time as an inlet for weak gas liquor or water. The revolving brushes or plates dip into the water and carry it around, so that the gas as it passes from one compartment to the other comes in contact with large surfaces of water, which aids greatly in the purification of the gas, i. e., in the absorption of the ammonia carried along with the gas. The reason for running in the water or weak gas liquor is that a weak gas liquor absorbs traces of ammonia much more readily from a nearly pure gas than would a nearly saturated ammoniacal solution.

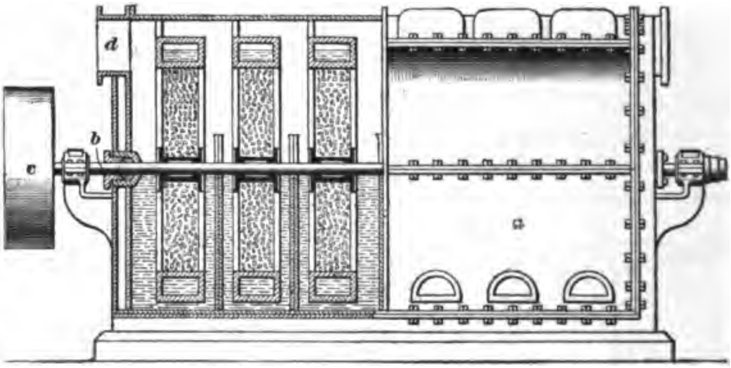

FIG. 15

34. Scrubbers.—The gas on leaving the washer is usually conducted to a scrubber. A **scrubber** is shown in Fig. 16, and consists of an upright iron cylinder *a*, in which are several layers of coke, brush, and excelsior. Water or weak gas liquor is forced through pipe *b* to the top, from where it trickles down through the coke and other material. The gas enters through *c* and on its ascent meets the water on its downward way. An extremely large wet surface is thus exposed to the gas, and in this way the last traces of ammonia are removed. The gas escapes through the outlet pipe *d*. Where two scrubbers are used, weak liquor is used in the first one and water in the second one.

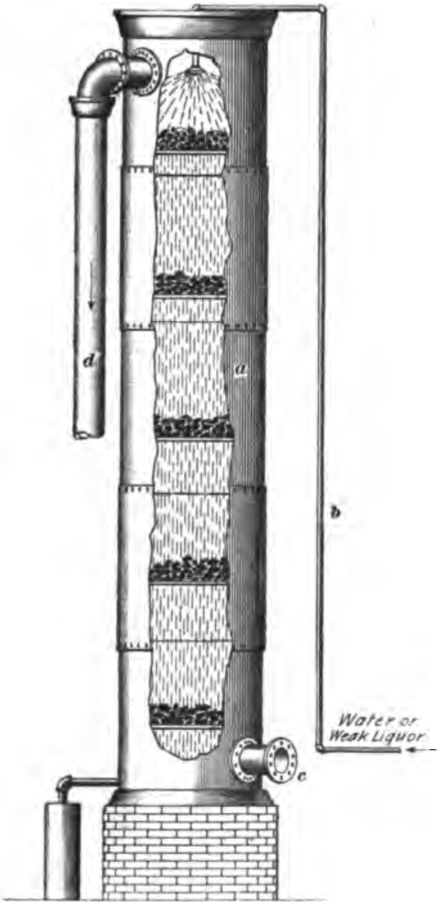

FIG. 16

35. Purifiers. The gas is now passed forward to the **purifiers**, where the remaining hydrogen sulphide, and, according to the nature of the purifying material, the rest of the carbon dioxide, is removed. Fig. 17 shows this apparatus, which consists essentially of a set of two or more pans, nearly square and about one-quarter as deep as they are long. Two or more layers of wooden gratings are placed in these boxes, on which layers of the purifying material are placed. The inlet and outlet are so placed that the gas enters below the lowest layer of the purifying agent and leaves above the top layer, thus having passed through the entire mass. A removable cover is provided, the sides of which rest in a water seal that runs entirely around the box, so that the cover is gas-tight when in place.

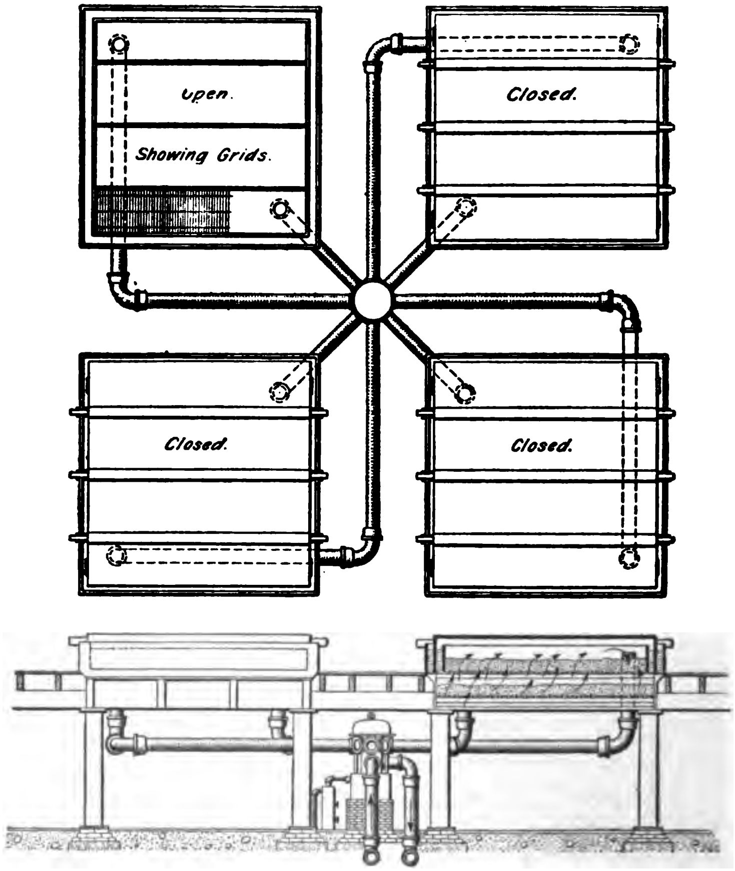

FIG. 17

In a large number of works the pans are arranged in groups of four (see Fig. 17), and are so connected that three are always in use at one time, the fourth being cut out so that it can be opened and refilled with fresh purifying material.

36. Center Seal.—The connection between the pans may be controlled by a number of valves. As a rule, however, a so-called **center seal,** shown in Fig. 18, is more commonly used. This center seal may be called a compound valve, in which passages are so arranged that for each quarter-turn it makes three pans are connected in series, while the fourth one is cut out. Thus, if the pans be numbered *1*, *2*, *3*, *4*, respectively, and *1* is cut out, *2*, *3*, and *4* will be in use, the gas passing through *2*, from there to *3*, and so on. Then, if the center-seal cover is turned one-quarter around, *2* is cut out and *3*, *4*, and *1* are placed in use. The gas reaches pan *1* at last, which is full of fresh material, and care is taken to see that the two pans through which it has previously passed have completely removed the hydrogen sulphide. The pan last put on in every case acts only as a safeguard to prevent any possible chance of impure gas getting through.

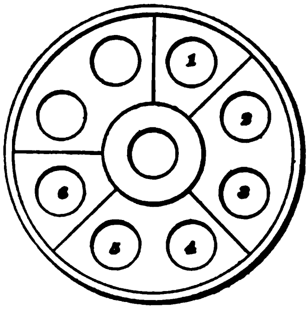

FIG. 18

37. Purifying Material.—The purifying material may be either lime or iron oxide. Up to within a few years lime was used exclusively. Slaked lime moistened with water is placed in layers from 6 to 12 inches thick, and the gas is passed through these layers. Carbonic acid and hydrogen sulphide are absorbed, the tendency being, however, for the carbonic acid to drive out the hydrogen sulphide from the first pan, so that it is absorbed by the next pan ahead. The lime in this is thus sulphided, and in this state serves to remove the bisulphide of carbon and other compounds that exist in small quantities in the gas. The third pan is used merely to prevent any hydrogen sulphide getting through the purifiers. It is customary to change pans when the gas begins to show hydrogen sulphide at the outlet of the second pan. This is detected by allowing a stream of gas from a cock to blow against moist lead paper, which turns brown if any hydrogen sulphide is present in the gas. From 10,000 to 15,000 cubic feet of gas are usually purified per bushel of lime, the lime being measured before it is slaked. The lime should be moist enough to cake readily in the hand when pressed. After the lime is spent it is practically of no value, although it is sometimes used as a fertilizer.

For this reason, oxide of iron has now almost entirely replaced it. This removes the hydrogen sulphide, but neither the carbonic acid nor other sulphur compounds. The amounts of the latter that escape the works, in most cases are so small that they can be safely neglected. The only effect that the carbonic acid has on the gas is to diminish the candlepower. It is said that 1 per cent. will reduce the illuminating power of gas about 1 candlepower, but most companies prefer to enrich the gas slightly when necessary, rather than to go to the expense of using lime. Oxide of iron may be procured either as natural hydrated oxide, sometimes mixed with sawdust or small shavings, or it may be manufactured by mixing thoroughly rusted cast-iron borings with sawdust. The borings when secured must be free from oil or grease, and may be rusted most simply with salt and water, or with weak gas liquor where that is at hand. The borings and sawdust (preferably white pine) should be mixed dry and shoveled over several times to insure a thorough mixture, and should be spread out on a cement or other hard, smooth floor in a layer not over 1 foot thick. The weak gas liquor should then be added, and the mass turned frequently with a shovel. It will begin to heat at once, on account of the rapid oxidation that will take place, and care must be taken to keep it exposed to the air by frequent turning, and to avoid large heaps, as otherwise it will take fire. It is essential that every particle of iron in the entire mass be covered with a coating of oxide, or rust, as otherwise the action of the gas on the iron will form iron carbonyl. This is a very volatile liquid, and will be consequently carried forwards with the gas. As it is decomposed by heat at 600° F., it will cause great trouble by the clogging of tips with a deposit of iron and by the discoloration of Welsbach mantles.

Oxide of iron is generally placed in the purifying pans in layers about 18 inches deep, two such layers being usually placed in each pan. About 10,000 cubic feet can be purified per bushel of oxide.

38. The action of the iron oxide may be expressed by the two equations:

$$Fe_2O_3 \cdot H_2O + 3H_2S = Fe_2S_3 + 4H_2O \qquad (1)$$

$$Fe_2O_3 \cdot H_2O + 3H_2S = 2FeS + S + 4H_2O \qquad (2)$$

Reaction 1 is considered the chief reaction taking place. The advantage of the hydrated ferric oxide over lime is that the mixture of Fe_2S_3 and FeS can be taken out of the purifier and exposed to the air, whereby it is revivified in accordance with the following equations, in which the water is not expressed:

$$Fe_2S_3 + O_3 = Fe_2O_3 + S_3 \qquad (1)$$

$$FeS + O = FeO + S \qquad (2)$$

The FeO becomes converted into Fe_2O_3 on further exposure. The oxide can be used repeatedly until it is *spent;* i. e., until it contains so much sulphur (50 per cent.) and other impurities as to be inoperative. The revivification can be effected simultaneously with the purification, and the labor of removal from the purifier avoided, by the admission of 2 per cent. of air into the gas before it enters the purifier. This, however, is objectionable, as it introduces nitrogen at the same time.

The spent oxide possesses some value on account of the sulphur it contains, which may be recovered by burning it in the manner of pyrites. As the hydrated ferric oxide also removes HCN and $HSCN$, it is, when spent, valued chiefly on the European continent for the Prussian blue and sulphocyanides that it is capable of yielding.

GAS METERS AND GAS HOLDERS

39. Wet Station Meters.—From the purifying house the gas passes to the **station meter,** usually a very large meter of the wet type, shown in Fig. 19, where the gas is measured. The reading of the station meter is usually taken every 12 hours, at the time the shifts of stokers change, and a check is thus placed on the work of each shift. The weight of coal charged being known, the amount of gas that should be produced can be roughly figured. In some large works the meter is read as often as every hour. It is of the greatest importance that the station meter should be as near correct as possible, and it should be tested at intervals to be sure of this.

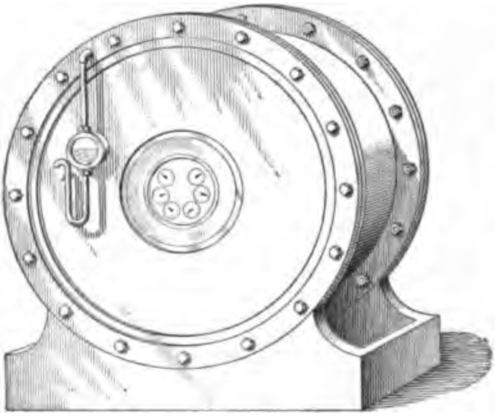

FIG. 19

40. Testing Wet Station Meters.—The meter room should be carefully ventilated while the test is being made, and no light or flame of any kind should be allowed in the building. The following points are to be observed in the test: (1) Are there any leaks in the drum? (2) How many cubic feet of gas are passed for each revolution of drum? (3) Does the index register correctly the quantity of gas passed through the drum?

The glass plate in front of the meter dial should be removed, the meter then by-passed, and a pointer attached to the spindle that sticks out through the meter head. The meter may then be started again, and an observation taken of the amount of gas passed during 10 revolutions of the pointer. From this the volume of gas passed for each revolution of the drum can be easily calculated. The meter should then be again by-passed and the valves closed on the inlet and outlet, which are in the rear of the meter. The inlet pipe should be tapped on both sides of the valve, with a 2-inch tap if possible, and a large meter of any size, from a 45-light to a 100-light, should be set so that the inlet of the meter connects with the tap made in the inlet pipe, on the inlet side of the valve, while the outlet of the meter connects with the tap in the inlet pipe on the opposite side of the valve. A cock should be placed at the inlet connection of the meter, and a **U** pressure gauge set at both the inlet and outlet. The cock on the meter connection should be closed. The outlet pipe of the station should also be tapped, and a pipe controlled by a cock should be run to the outside of the meter house. The gauge at the inlet should be removed for a short time, so that the station meter may be at atmospheric pressure inside. The manhole on the station meter should be opened and the drum blocked so that it cannot turn. The manhole must then be closed and the gas turned on the meter, through the test meter, until the seal in the station meter begins to blow. The blowing pressure should be noted on the **U** gauge at the outlet of the test meter. The meter cock should then be closed, and the **U** gauge watched to see if the pressure falls. If this is the case, the section of the drum that is being tested leaks.

As there are usually eight sections, it is necessary to make as many different tests in the same manner, the drum being turned one-eighth revolution each time. The number of revolutions of the points, compared with the number made by the drum, should be noted, and should usually be the same or just double the number. Note that the water is just at the working level during this test.

41. If the drum leaks anywhere, it must be taken out and repaired before going any further with the section-meter test. The drum should now be released so that it can turn, and the meter cock on the test meter opened so that as much gas passes as is consistent with the test meter. Both meters will be passing gas, and the index of the test meter must be read at the instant the pointer on the station meter passes a given point, and read again when the pointer comes around again to the given point. The quantity of gas passed by one revolution of the station meter is thus measured. Several revolutions should be made, and an average taken, to secure accurate results.

Count the number of teeth in the gear-wheel connecting the spindle with the index gear, and figure up how many cubic feet should be registered by one revolution of the drum.. This figure should agree very closely with the first calculation made in the station-meter test. If the figures are about the same, the meter is correct. If the result of the first calculation shows a larger registration of gas than was actually passed, the meter is fast; if the contrary, it is slow. If the meter is not correct, the gear-wheels must be changed. Very small errors may be corrected by changing the water level a little, raising it if the meter is slow and lowering it if the meter is fast. The water in the station meter and the air in the meter house should be as nearly as possible at the same temperature during the test.

The test meter should be carefully tested by a prover under the same conditions and at the same rate as during the station-meter test. This condition may be easily obtained by maintaining the same difference of pressure at inlet and outlet that was observed during the station-meter test.

42. Proportional Station Meters.—In some gasworks **proportional station meters** are used. These meters have two valves set in parallel that have some definite proportion in size, such as 1 to 100. The valves are worked on the same set of levers, so that no matter whether each valve be wide open or only cracked, the proportion of the openings is always the same. The gas is divided into two streams, as it were, part going through the large valve and part going through the small valve. There is a regulator that governs the pressure of the gas under the large valve, so that it is always the same as at the small valve. The gas going through the small valve is metered by a small meter known as a **tally meter,** and the index gear of this meter is geared up 100 times, if the proportion of the valves is 100 to 1, so that it gives a direct reading of the gas passed through the meter. The proportional meter is much cheaper than the ordinary wet meter, and with careful watching will do very good work.

43. Testing Proportional Station Meters. — Proportional meters are tested as follows: A **U** gauge is connected by one leg to the inlet side of the main valve, and by the other to the inlet side of the tally meter. The two pressures should balance themselves, so that the water in the gauge stands even in both legs, or nearly so. If this is not the case, attention must be given to the apparatus governing the pressure under the main valve. The pressure being found to balance, the meter is by-passed, and the tally meter is taken off and tested on a prover, about the same rate of flow being maintained at the prover as is customary in the actual use of the meter. If the tally meter is found to be correct and the pressures balance, the proportional meter will be correct, provided the valves have been properly proportioned.

44. It is sometimes convenient to check the station meter by allowing gas to pass through it for a considerable period of time into a holder from which no gas is being sent out. The height of the holder is taken at the beginning and end of the period, as are also the meter readings. This test should be made on a cool, cloudy day, if possible, and it is well to lower a slender thermometer on a cord through the plug hole in the top of the holder, stopping the escape of gas by a cork. The thermometer should be well down in the holder, and should be drawn up and read at frequent intervals. A thermometer on the meter should also be used, and the volume of gas received in the holder may be very actually checked against the meter reading after the proper corrections have been made for the temperature.

All station meters should be equipped with thermometers, and a record of the temperature taken at frequent intervals, as well as the reading of a barometer. The volume of gas is affected by both the changes in barometric pressure and temperature, and in practical work all measurements of gas in large quantities are corrected to normal temperature and pressure.

45. Gas Holders.—From the station meter the gas goes to the **holder,** or **gasometer,** to be stored until it is needed. The storage capacity of gasworks should be equal to the maximum quantity of gas consumed in 24 hours. The gas holder is a cylindrical vessel closed at the top, which is known as the roof, and is open at the bottom. Holders are made of sheet iron of varying thickness, according to the size of the vessel. The holder is rather less in diameter than, but of the same depth as, the tank into which it flows, and it is guided by rollers that are attached to its upper and

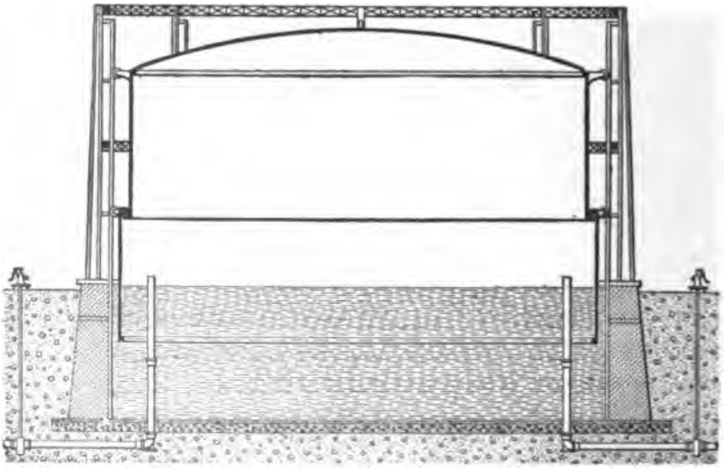

FIG. 20

lower edges at intervals along its circumference. These rollers work against suitable guides fixed in the tank, and on a series of columns, or standards, placed at equal distances around the top edge of the tank, so as to allow the holder to rise and fall at ease. A gas holder is shown in Fig. 20. An inlet and outlet pipe extend through the bottom of the tank, up above the water level through which the gas is admitted to the inside of the holder and removed from it. If gas is pumped into the holder under pressure, the space between the water and the holder is soon filled and the holder begins to rise. It is guided as it rises by wheels that bear against iron columns set at intervals on the edge of the tank. The holder is thus free to rise or fall, according as gas is admitted or taken away, the water meanwhile acting as a seal to prevent the escape of gas.

Holders are frequently built in several sections that telescope into one another, being joined by a grip with a water seal, so that there is no leak at the joint. Such a grip and cup is shown in Fig. 21. Holders are made of all sizes, from small, single-lift holders of a capacity of only a few thousand feet, to those with several lifts capable of holding as much as 10,000,000 cubic feet. The weight of the holder fixes the maximum pressure at which gas may be delivered.

46. Action of the Gas Holder.—The action of the gas holder by which it rises and falls is as follows: Supposing the holder to be empty of gas and filled with water; then, on allowing gas to enter the inlet pipe, as there is space between the surface of the water and the roof of the holder, and as the top inlet pipe is above the level of the water in the tank, the gas fills this space; as it possesses a certain amount of pressure, this pressure is exerted on the surface of the water and on the inside of the roof, thus causing the holder to rise. The water in the tank

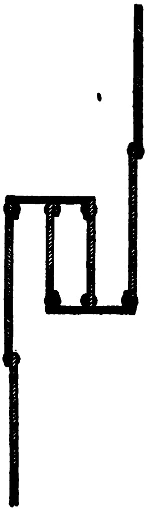

FIG. 21

may be said to serve three purposes: It offers the necessary resistance to the gas that enables the holder to rise; when the holder is falling, it is the medium by which the gas is expelled; it also prevents the gas from escaping from the holder.

47. Governors.—The gas as it comes from the holder passes to a **governor,** from which it is delivered to the street mains. It is the office of the governor to reduce the holder pressure to a pressure just sufficient to supply the consumers at the points farthermost from the works. Governors may be divided into two classes: those that may be set to maintain any desired pressure regardless of the volume passed; and those that automatically increase the pressure as the volume passed increases, and decrease it as the volume decreases. Such is the Connelly governor shown in Fig. 22. In the older style of governors, the weights are added by hand to the float, to make the governor give any increase in pressure that may be necessary. In the Connelly governor, mercury is transferred automatically to give the weight required for an increased pressure.

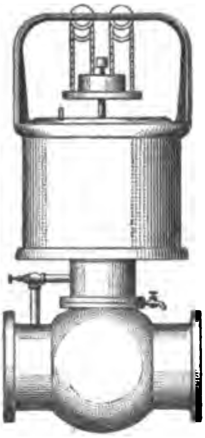

FIG. 22

From the governor the gas passes out through the street mains to the consumers.

AMMONIACAL LIQUOR

48. Ammoniacal liquor as it comes from the washer is usually of about what is known as 4-ounce strength. This means that there is enough ammonia in 1 gallon of the liquor to neutralize 4 ounces of normal sulphuric acid. The custom of valuing gas liquor in this way is an English one; it is more common in this country to value the liquor by either the percentage or weight of ammonia present in the liquor.

The strength of weak ammoniacal liquor may be roughly estimated by the use of a Twaddell No. 1 hydrometer. The hydrometer is allowed to float in a glass vessel containing the liquor, and the number of divisions on the stem above the level of the liquor are read off. With weak liquor, each degree is about equal to 2 ounces, so that, if 2 degrees is shown by the hydrometer, the liquor may be considered to be about 4-ounce strength. Four-ounce liquor contains approximately 1 per cent. of ammonia.

The ammonia is present in the liquor partly as free ammonia and partly as fixed ammonia. The free ammonia may be driven off by heating the liquor to above 180° F., but the fixed ammonia cannot be driven off by heat unless some alkali, such as lime, caustic potash, or caustic soda, is used. The weak ammoniacal liquor contains such a large percentage of water that it must be distilled before it can be marketed with profit. Many forms of distilling apparatus are in use, but the same principle is observed in each case. The liquor must be heated in order that the free ammonia may be driven off, and when the amount of fixed ammonia is sufficient to pay for the trouble in working it up, milk of lime, a solution of slaked lime and water, is added to liberate the fixed ammonia. The ammonia liberated by the heat is condensed in cooling coils, and either received in a vessel containing sulphuric acid, in which case ammonia sulphate is formed, or more commonly a sufficient amount of steam to form a strong ammoniacal liquor containing from 15 to 20 per cent. of ammonia is condensed with it. The waste liquor should be smelled frequently, to see if there is any ammonia escaping the still, and at intervals it should be analyzed. This concentrated liquor is sold to chemical factories. The liquor must be above 15 per cent. to find a ready market, and it is found, where liquor of more than 20 per cent. is made, that the carbonate of ammonia and other salts are apt to form in the condensing coils and block up the apparatus, so that it is impracticable to turn out liquor of more than 20 per cent. from stills commonly used in gasworks.

49. Ammonia Stills.—The first stills used were of the intermittent type. That is, they were filled up with liquor when cool, and a fire was then built under them and the ammonia distilled off. After this they were allowed to cool down, and the waste liquor was drawn off and a fresh charge put in. The modern stills are usually of the continuous-operation type. Live steam is injected to heat the liquor, which is fed in continuously, while the overflow of the waste is arranged so that the waste liquor is discharged after the ammonia has been all driven out.

Fig. 23 is a representation of a modern ammonia still. *A* represents the fixed still containing the crude ammoniacal liquor; *B*, the volatile still from where the ammonia gases, both free and combined, pass to *C* and *D*, the first and second condenser; leaving the latter, they pass to the first absorber *E*, and any escaping gases are caught in the second condenser *F*.

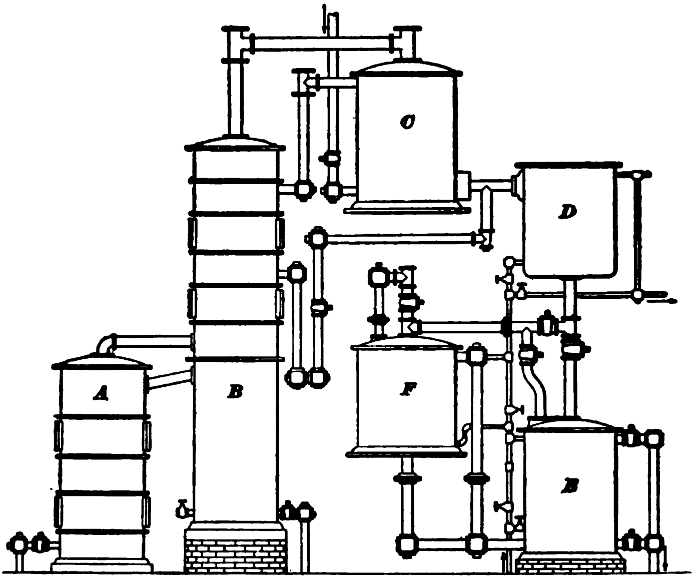

FIG. 23

50. The Twaddell hydrometer is used as a rough indicator of the strength of the concentrated liquor. Table IV shows the specific gravity, weight per cubic foot, weight per gallon, and ounce strength of ammoniacal liquor of different degrees Twaddell.

TABLE IV

Degrees Twaddell	Specific Gravity and Weight Per Cubic Foot in Ounces Avoirdupois	Weight Per Gallon. Pounds	Ounce Strength	Degrees Twaddell	Specific Gravity and Weight Per Cubic Foot in Ounces Avoirdupois	Weight Per Gallon. Pounds	Ounce Strength
0	1,000.0	10.000	0	12½	1,062.5	10.625	25
½	1,002.5	10.025	1	13	1,065.0	10.650	26
1	1,005.0	10.050	2	13½	1,067.5	10.675	27
1½	1,007.5	10.075	3	14	1,070.0	10.700	28
2	1,010.0	10.100	4	14½	1,072.5	10.725	29
2½	1,012.5	10.125	5	15	1,075.0	10.750	30
3	1,015.0	10.150	6	15½	1,077.5	10.775	31
3½	1,017.5	10.175	7	16	1,080.0	10.800	32
4	1,020.0	10.200	8	16½	1,082.5	10.825	33
4½	1,022.5	10.225	9	17	1,085.0	10.850	34
5	1,025.0	10.250	10	17½	1,087.5	10.875	35
5½	1,027.5	10.275	11	18	1,090.0	10.900	36
6	1,030.0	10.300	12	18½	1,092.5	10.925	37
6½	1,032.5	10.325	13	19	1,095.0	10.950	38
7	1,035.0	10.350	14	19½	1,097.5	10.975	39
7½	1,037.5	10.375	15	20	1,100.0	11.000	40
8	1,040.0	10.400	16	20½	1,102.5	11.025	41
8½	1,042.5	10.425	17	21	1,105.0	11.050	42
9	1,045.0	10.450	18	21½	1,107.5	11.075	43
9½	1,047.5	10.475	19	22	1,110.0	11.100	44
10	1,050.0	10.500	20	22½	1,112.5	11.125	45
10½	1,052.5	10.525	21	23	1,115.0	11.115	46
11	1,055.0	10.550	22	23½	1,117.5	11.175	47
11½	1,057.5	10.575	23	24	1,120.0	11.200	48
12	1,060.0	10.600	24	25	1,125.0	11.250	50

51. It will be seen from this table that the weight of the liquor in pounds avoirdupois per gallon is obtained by simply placing the decimal point after the first two figures of the number representing the specific gravity. Thus, for instance, liquor of 1,025 specific gravity weighs 10.25 pounds per gallon.

If a more accurate determination is required, a volumetric analysis of the free and combined ammonia should be made (see *Quantitative Analysis*).

Ammoniacal liquor is usually sold by weight, the value being based on the weight of ammonia in the liquor. Thus, if a tank of concentrated liquor weighs 1,000 pounds, and the liquor contains 15.5 per cent. of ammonia, its value at 7 cents per pound would be $10.35.

MANUFACTURE OF GAS

(PART 2)

WATER GAS

INTRODUCTORY

1. Water gas is a mixture of hydrogen and carbon monoxide. It is manufactured commercially by the contact of steam with incandescent carbon in the form of anthracite coal or coke. The steam is decomposed, the hydrogen being separated from the oxygen. The oxygen takes up carbon from the coal or coke and forms carbon monoxide, along with a small amount of carbon dioxide. The resultant gases from the contact of steam with incandescent carbon are then mainly hydrogen and monoxide, chemically separate but mechanically mixed together. This is what is called *blue*, or *uncarbureted*, water gas. It burns with a non-luminous flame and is consequently useless for lighting purposes except in incandescent lamps of the Welsbach type. In actual practice this water gas is always enriched with oil gas, which furnishes the hydrocarbons necessary to make a luminous flame. The oil gas was made separately in many of the older forms of apparatus, but it is now commonly produced in the same apparatus in which the water gas is made. The only impurity found in water gas that must be removed is hydrogen sulphide, which is formed from the sulphur always present in greater or less amount

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in the coal or coke and sometimes in the oil. The hydrogen sulphide is removed by purification with lime or iron oxide in the same way that the purification of coal gas is accomplished.

Carbon dioxide, which is formed by either imperfect contact of the steam with the incandescent carbon, or because the temperature of the carbon is too low, is not a dangerous impurity, but is merely an inert gas incapable of combustion. It, however, absorbs heat when the gas is burned, and is consequently injurious to the heating and lighting power. It can be removed by purification with lime, but this is not necessary if the generating apparatus is handled properly, as the quantity made will be very small. No ammonia is produced.

2. **Composition of Purified Water Gas.**—The following is a volumetric analysis of a sample of purified water gas:

Hydrocarbon vapors	1.2
Carbon dioxide	3.0
Heavy hydrocarbons	12.6
Oxygen	.4
Carbon monoxide	28.0
Hydrogen	31.4
Methane	20.2
Nitrogen	3.2
Total	100.0

3. Water gas requires from 30 to 40 pounds of coal or coke per 1,000 cubic feet of gas made, and from 4 to 5 gallons of oil, depending on the candlepower required. Usually between 5 and 6 candlepower is obtained from each gallon of oil used. The specific gravity of 24-candlepower water gas is about .625, air being taken as unity.

Pure uncarbureted gas has no perceptible odor, but the carbureted gas has an odor fully as strong as coal gas. This is mainly due to the hydrocarbons from the oil that is used for enriching.

MANUFACTURING PROCESS

4. Almost all water-gas machines now in use are modifications of the **Lowe** type. The Lowe type of apparatus consist of a *generator*, where the blue water gas is produced, and a *superheater ;* or a *carbureter* and a superheater, where the oil is vaporized and mixed with the blue water gas. The generator is a circular steel shell, the height of which is about one and a half times its diameter. It is lined with a double lining of firebrick blocks and is provided with grate bars at the lower end and with air-tight doors at the top, where the coal is charged in, and at the bottom, where the clinkers are taken out. There are also connections for the escape of the gas and for the proper supply of steam and air.

5. The capacity of any generator depends largely on the grate area and may be figured at a minimum of 20,000 cubic feet per square foot of grate surface per 24 hours. In some cases the generator connects directly to the superheater, but in most machines a carbureter is placed between the two. Both carbureter and superheater are steel shells about the diameter of the generator. The carbureter is usually about the same height as the generator, and the superheater may either be about the same height as the generator or about one and a half times its height. The latter is the more usual construction. Both carbureter and superheater are lined with firebrick and supplied with valves for the admission of air. They are also filled with firebrick set on edge in rows, with a space of about 1½ inches between each row. The rows of brick in each tier are run at right angles to those below them, and the brick are so placed that each row comes directly over the space left between the two rows of brick running in the same direction in the second tier below. The brick filling up the carbureter and superheater are called *checker brick.*

6. On top of the superheater the stack valve is placed. This valve opens into a stack, and when it is closed any gas that is produced in the apparatus is forced out through a take-off pipe down into a round iron box called the seal.

The seal connects with a scrubber, and the scrubber connects with a condenser. From the condenser a pipe usually

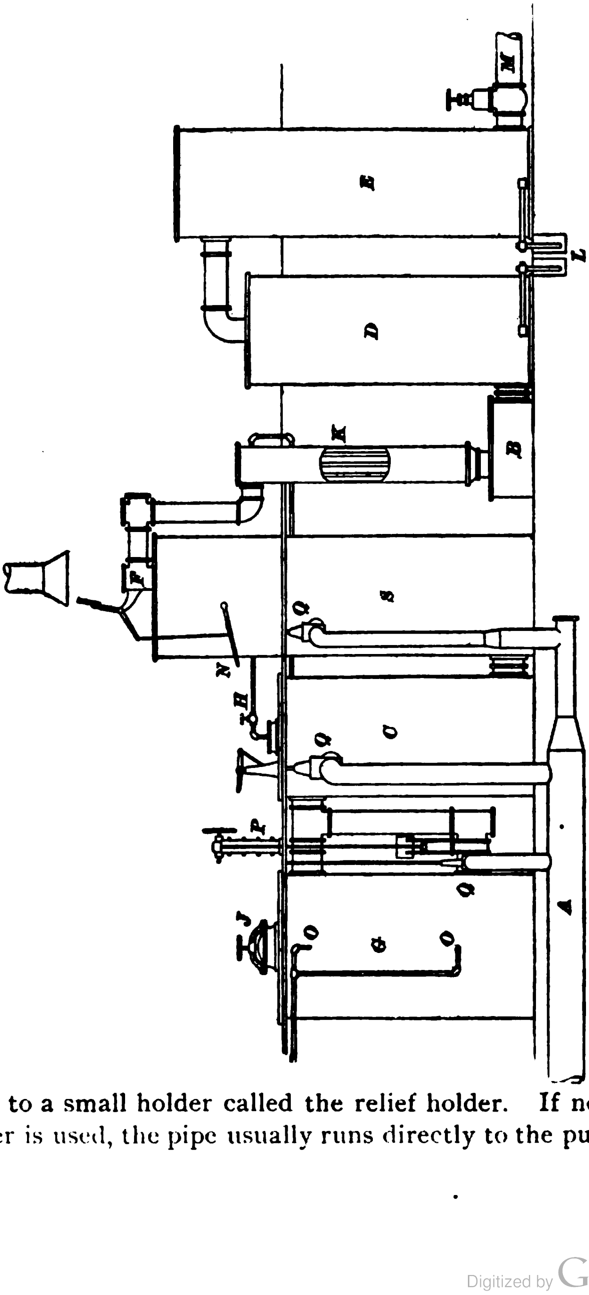

FIG. 1

leads to a small holder called the relief holder. If no relief holder is used, the pipe usually runs directly to the purifiers.

Sometimes a wash box similar to the ammonia washer is placed on the line to remove all the oil and tar from the gas before it gets to the purifiers.

7. **Water-Gas Apparatus.**—Fig. 1 shows the complete apparatus, where *G* is the generator, *C* the carbureter, *S* the superheater, *B* the seal, *D* the scrubber, *E* the condenser, *F* the stack valve, *H* the oil valve, *J* the cooling branch, *K* the oil heater, *L* the seal pots, *M* the gas outlet to the relief holder, *A* the air pipe from the blower, *N* the stack-valve lever, *O*, *O* the up-and-down steam valves, *P* the up-and-down run valves, and *Q*, *Q* the air-blast valves. The generator is provided with a steam pipe below the grate bars, and if it is what is known as an up-and-down run machine, there is also steam connection above the level of the fuel bed. The up-and-down run machine has also a gas outlet both above and below the fuel bed, each outlet being

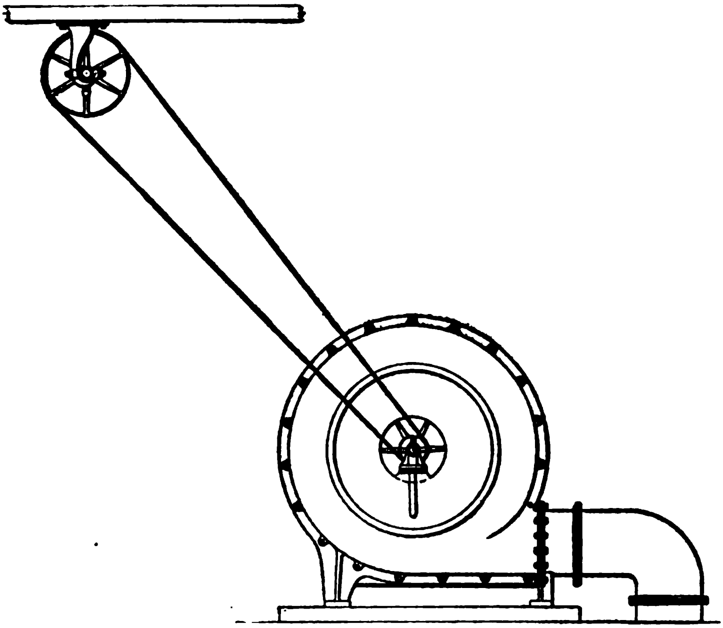

FIG. 2

controlled by a valve. Large air pipes controlled by suitable valves enter the generator below the grate-bar level, the carbureter near the top, and the superheater near the bottom. Air is supplied to the machine through these from a blower, usually of the centrifugal type, driven by an engine, as shown in Fig. 2.

8. Drying Water-Gas Apparatus.—When a new set of water-gas apparatus is to be fired up, it should be dried out as follows: Cover the grate bars with 4 or 5 inches of cinders and fill up the generator with shavings and dry wood. Leave the generator coaling branch open and light the fire on the grate. When a good fire is started, close the lid on the coaling door and let the fire draw through the carbureter and superheater. A small steam jet placed so that it points up the stack will create a draft and remove the wood smoke that is otherwise apt to fill the generator room. When the wood fire has burned down somewhat, add coal or coke until a fire about 2 feet thick is built; then check the draft by partly closing the lower fire-doors. Four or five days should be allowed to dry out a green set, but if necessary, it can be fired up under blast and put in operation in 12 hours. In drying out, the fuel should be kept about 2 feet deep and the fire shaken up at intervals with a bar from the side doors. When coaling, open the generator lid a little and apply a piece of lighted paper before opening any farther, as otherwise a violent explosion may result. When the shells feel warm, the apparatus is ready to blast up for the first heat.

9. Starting Water-Gas Apparatus.—An apparatus that has been in use and has been laid off can be fired up very quickly without any danger of damage. If hot coke is at hand, the generator can be partly filled with it and then filled with cold coke or coal and the blast applied. If no hot coke is at hand, a wood fire must be built and allowed to burn freely, and the generator may then be fully charged with coal. In either case, when the generator is brought up quickly to a working heat, it is well to take one run off the fuel before opening again to coal, as otherwise a violent explosion is liable to occur when the coaling door is opened.

10. When ready to put on the blast, either on a green set that has been warmed up or an old set, see that the generator is filled with fuel almost to the level of the take-off pipe. See that the lower fire-doors and the ash-pit door are firmly clamped shut. In old machines, where the doors are warped so that they are not tight, use Dixon's graphite pipe-joint compound No. 695. This is far better than the paint, etc. frequently used.

Put the blast on the generator by opening the blast valve. The blast pressure under the grate should give a pressure of at least 10 inches of water. If the blast is continued for more than ½ hour, the steam valve on the steam line that runs under the fire should be opened a little. This will prevent burning the grate bars and the formation of clinkers.

When a flame can be seen through the sight cock in the top of the generator, raise the carbureter blast valve a little and look into the sight cock on top of the carbureter. If a blue flame is not seen, shut off the carbureter blast and try again in a few minutes. When the carbureter lights properly, increase the blast slowly.

When the carbureter brick, seen through the bottom sight cock, shows a red heat, reduce the carbureter blast and open the superheater blast valve a little, keeping watch for a blue flame through the lower superheater sight cock. The superheater may sometimes be lighted more readily by one man thrusting a red-hot pipe through the bottom sight cock, while another man raises the blast valve a little.

When the checker brick at the bottom of the superheater show a bright-red heat, the blast on it should be increased to throw the heat towards the top.

The first run, or make of gas, may be taken off at this point. The heat in the top of the superheater will come up while the set is working.

11. Working the Apparatus.—To make a run, shut off (1) superheater blast, (2) carbureter blast, (3) generator blast. Open the steam valve one-half turn. Then close the stack valve, increase the steam to the amount found desirable, open oil valve, and start oil pump. Regulate the feed of oil by a valve on the oil line, allowing the pump to run at a fixed speed. Make the oil feed such that all the oil will have been put in at least 1½ minutes before the end of the run. The feed should be a little faster the first half of the run. When, after several runs, the rate of production of the machine has been observed, the amount of oil can be regulated so that the desired number of gallons per thousand is put in. The amount of steam to be used can be regulated by the amount of carbonic acid in the gas. If the fire is kept in good condition, the carbonic acid should not exceed 3 per cent. when coal is used as fuel or 5 per cent. when gas-house coke is used. If the gas shows more than the above amount of carbonic acid, the steam supply should be reduced.

12. After about 5 or 6 minutes' run, shut off the oil, and 1 or 2 minutes later shut off the steam and open the stack valve. The blast valves should then be opened, generator first, carbureter second, and superheater third. About 8 minutes' blow and 8 minutes' run will be found a good average for coal and about 4 minutes' blow and 7 minutes' run for coke. The operator must, however, vary his runs to suit the machine, blowing longer when the heats are not high enough. When the superheater becomes too hot, especially when the set is run continually, it is frequently necessary to leave the superheater blast valve down and blast into the carbureter and generator only. The oil sprays should be examined once each day, whether they are of the rotary or the injector type, as they frequently get stopped up.

13. The generator should be coaled about every six runs when coal is used and about every four runs when coke is used. The coaling should be done just after a run, but care should be taken to open the generator blast valve for a few seconds and then to close it before opening the door on the coal-hole branch. All the foregoing description applies to the operation of what is known as a **base steam machine.**

14. A **reverse steam machine** is operated in the same manner, except that at every second or third run, the take-off pipe above the fuel bed is closed by the proper valve, and the valve on the lower take-off pipe below the grate bars is opened. Steam is then admitted through a pipe coming in above the fire and gas is made down through the fuel bed instead of up through it. By this means a more even temperature of the fuel bed is maintained and a resultant increase yielded with a lower percentage of carbonic acid.

At the end of the run the top valve must be opened and the lower one closed again before the blast is put on.

15. Cleaning the Fire.—The fire must be cleaned about every 6 hours with a base steam apparatus and about every 12 hours with a reverse steam apparatus. Before cleaning, the door on the coal-hole branch should be slacked off and lighted. The ash-pit door should be opened, and after this the door on the coal-hole branch may be closed. The cleaning doors on the sides of the generator should then be opened and the clinkers and ashes drawn out with an iron hook.

The ashes that fall through the grate bar may be drawn out through the ash door. Be careful to clean out all loose stuff before cutting the clinkers. After the loose stuff is all removed, cut out the clinkers. If the fire falls before the clinkers are removed, cut right across the surface of the grate with heavy slice bars.

Next, clean all doors and shut them up tight; then bar the fire down from the coaling door. Cut down around the outside of the fire to loosen all clinkers and loosen up the fuel bed all over with the bar. If there are any dead spots in the fire, cover them over with hot fuel. A large steel bar about 2 inches in diameter is necessary for this work. It should be sharpened on the end, and it will be found necessary in some cases to use a sledge hammer to drive it through the clinkers. Where the clinkers are very bad, their removal may be facilitated by dumping a barrel of oyster shells into the generator about 2 hours before cleaning.

When the fire has been thoroughly barred, fill up the generator with fuel as high as the take-off pipe. The coal or coke should be thrown in so that it fills the whole generator evenly. If it is merely dumped in the center, the steam when turned on will come up around the outside of the fire and the product will be poor and the volume of carbonic acid large.

With some cokes it is advantageous to ram the fuel bed around the outside edge with an iron pounder. After coaling up, put on the blast in the regular way. It will be necessary to blow considerably longer than usual after cleaning the fire.

16. When ready to shut down the apparatus, blow out the generator for 1 or 2 minutes and then shut off the air blast. Next, crack open the generator lid and light it. Then open the ash door and shut it against a piece of tin so as to leave a small crack that will allow enough air to enter to keep the fire alive.

When ready to start up again, close both doors and put on the generator blast and bring the machine to a working heat in the same way as heretofore described. The fire may be cleaned either before putting on the blast or after three or four runs are made. The latter is rather the best plan.

17. Precautions.—While running, a close watch should be kept on the character of the overflow of the seal. Where naphtha is used, if large amounts of oil come out, the heats of the carbureter and superheater are too low. Where crude oil is used, a considerable quantity of tar is produced.

If lampblack is observed in the seal overflow, the heats are too high and should at once be lowered, as when the heats are high enough to make lampblack, the candlepower of the gas is very materially lowered. Besides this, the lampblack is deposited on the checker brick, forming a non-conducting coat that reduces very much the oil efficiency of the carbureter.

18. Tar Separator.—The oil tar that comes from the seal and scrubber should be separated from the water and not allowed to flow away, as it is so near the gravity of the water that it may be carried a great distance and be the cause of considerable complaint. A convenient separator is a long box, either of wood or iron, arranged with baffle plates or partitions in such a way that the mixture of tar and water has to flow under one plate and over the next, as shown in Fig. 3. The partition should be cut away a little at the bottom to allow the tar to flow along. It may be either conducted by gravity to a tar tank or pumped out of the separator. This apparatus, if of sufficient size, will remove nearly all the tar.

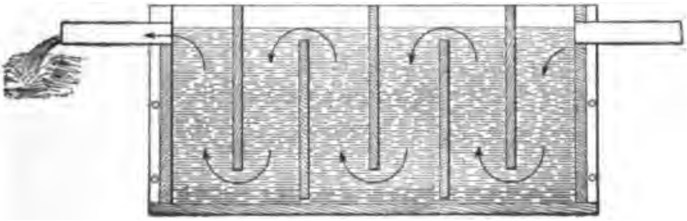

FIG. 3

As the oil and tar separate more readily when hot than when cold, the separator should be as near the seal as possible. If it must be placed at some distance, the pipe to it should be either buried in the ground or covered with some good non-conductive pipe covering. The separator itself should be provided with a cover.

19. A **coke filter** should be placed at the outlet of the separating apparatus. This is simply a long box filled with fine coke or screenings from the water-gas apparatus. The water is allowed to flow in at one end and pass down through the coke and out the other end. This filter will remove the light oil not taken out by the separator. When the filter begins to allow oil to escape, the coke should be taken out and a fresh lot put in. The coke that has been removed may be allowed to dry and then be used for boiler fuel.

20. A **pressure gauge** of the cup type should be provided for each piece of apparatus comprising a water-gas set. These gauges should be placed on a board where they may be readily observed by the operator, and connected by pipes to the inlets of the various pieces of apparatus. By observing these, the operator can at once note the location of any clogging that may occur. The most usual clogging occurs from lampblack deposits in the stand pipe that connects the gas take-off below the stack valve with the seal. Cleaning doors are always provided on the stand pipe. No light or flame of any kind should be allowed near the stand pipe when the cleaning doors are opened. The upper door should be opened first, and the take-off pipe, where it enters the superheater, stopped up with an old bag or some similar substance. The lower door may then be opened, and it is best to stop up the stand pipe below this door to prevent lampblack falling into the seal.

The pipe may next be cleaned out, pieces of pipe or iron rods being used to dislodge the lampblack, which will then fall to the lower door. After the pipe above the lower door is thoroughly cleaned, the bag or other substance stopping it below the doors may be removed, and the lampblack taken out down in the pipe where it dips into the seal. If much lampblack should fall into the seal, it should be opened and cleaned out at once. Before attempting any of this work, it is well to see that the valve between the water-gas machine and the relief holder is shut. This valve should be shut whenever the machine is shut down for more than an hour or two. Care must also be taken to see that this valve is opened before the first run is put on when the machine is started.

21. It must always be remembered in operating a water-gas set that the blast valves must never be opened before the stack valve has been opened, and that the stack valve must never be closed until the blast valves have all been closed.

22. Oil.—The oil that is used in making water gas is usually run through a heater, after it leaves the pump and before it enters the machine. In some cases this heater is simply several coils of pipe that are placed inside the stand pipe above the seal, where the oil is heated by the hot gas leaving the apparatus. In this arrangement, however, the lampblack, which soon covers the pipes, forms an insulating coat, preventing the oil from being heated properly. A better form of heater is a coil of pipe inside an iron drum or cylinder.

23. The exhaust steam from the blower engines and pumps is passed through the cylinder and heats the oil. The oil that is used in making gas must be accurately measured. This is accomplished in some cases by the use of a small tank provided with a glass gauge, so that the level of the oil can be read after each run. A scale reading gallons is placed alongside the gauge glass, and the gas maker, after having pumped up oil during the time he is blowing, reads the number of gallons on hand at the beginning of the run. The oil is then pumped out of the tank into the machine, and at the end of the run the gas maker again reads the number of gallons on hand. The difference between this reading and the first gives the amount of oil used.

24. When the oil is sprayed into the carbureter, or, as in some machines, into the generator above the fire, it is at once vaporized by the intense heat. In this condition, however, it would mostly condense again into oil and tar when the gas cooled down, and for that reason the checker brick are provided. In passing over the hot surfaces of the carbureter and superheater the oil is *cracked up* and fixed in the gas permanently, so that very little of it condenses out when the gas is cooled.

The checker brick are gradually coated with lampblack and become saturated with oil that burns to carbon, and after a while do not hold the heat and fix the oil properly. They should then be taken out and replaced by new brick.

To do this it is necessary to cool off the machine and open the manhole doors that are provided on the sides of the carbureter and superheater. The length of time a machine may be run without renewing the checker brick varies with the kind of oil used and with the skill of the operator in keeping the heats neither too hot nor too cold. In machines that are run continually, the carbureter should be rebricked about every 6 months where naphtha is used, and about every 3 months where gas oil or crude oil is used.

25. Firebrick Lining.—When a water-gas apparatus is let down for repairs or rebricking, careful examination should be made of the inner firebrick lining of the generator. The parts that usually wear out first in the lining are the brick around the cleaning doors. These should be renewed if necessary, for, if the intense heat of the fire reaches the ironwork around the doors, it will become red hot at once and crack, and in some cases burn out entirely. If the inner lining is much burned or worn, it should be entirely removed and a new one put in.

The generator is always arranged with a through course of brick near the top, so that the lower part of the inner lining can be entirely taken out without danger.

The joints in the new lining should be made very close, with as little fireclay between them as possible, and care should be taken that the blocks around the cleaning doors are put in so that they will not fall out if the cast-iron sleeves, usually provided, burn out, as they are sure to do.

Careful attention should also be given to the condition of the brick in the arch around the coal-hole branch. The ends of these brick when burned are apt to be knocked off by the bar and the coal when it is shoveled in, and this may let down the neck brick in the coal-hole branch. If the generator is run in this condition, the neck casting will be burned or cracked. When the arch bricks are not in good shape, a new arch should be put in.

26. Relief Holder.—Water gas should always be made in a **relief holder.** With coal gas a relief holder is not necessary, because the output is practically uniform, but in making water gas, large quantities of gas are produced at intervals of from 4 to 10 minutes, according to the length of the blow. If the machine is to be used without a relief holder, the purifying apparatus and meter provided for a machine capable of making, say, 200,000 cubic feet in 24 hours must be at least three times the size of the same apparatus for a relief-holder plant. When a relief holder is used, the gas rushes into it intermittently right from the machine, but is removed by an exhauster and passes through the purifiers and the meter at a uniform rate. In addition to this, it is found that the relief holder acts in some measure as a condenser, and the pans are not as likely to become clogged up with the deposited oil as when no relief holder is used.

There is also less back pressure on the water-gas apparatus during a run where a relief holder is used, and consequently better manufacturing results are obtained. The exhauster used in connection with the relief holder may be of about the same type as that used on coal gas, but it is not necessary to provide it with an automatic governor such as is used on the coal-gas exhauster.

27. Apparatus for the Determination of Carbonic-Acid Gas.—As the amount of carbonic acid in the water gas depends largely on whether the apparatus is properly handled or not, and as an excess of carbonic acid very seriously affects the illuminating power of the gas, it is convenient to have an apparatus by which the percentage of carbonic acid can be quickly and easily ascertained at any time. The fact that carbonic acid is readily absorbed by caustic potash is taken advantage of as follows: A solution of about 1 part by weight of potash to 3 parts by weight of water is prepared.

The absorption and measuring apparatus shown in Fig. 4 is placed in a convenient position, and the absorption pipette is filled with the potash solution. This pipette is best filled when made with small rolls of iron-wire gauze, as the absorbing surface is thus much increased. One branch of the three-way cock at the top of the measuring burette is connected by a capillary glass tube with the pipette. The joints are made of rubber tube. The measuring burette is made to hold 100 cubic centimeters, and is graduated to read

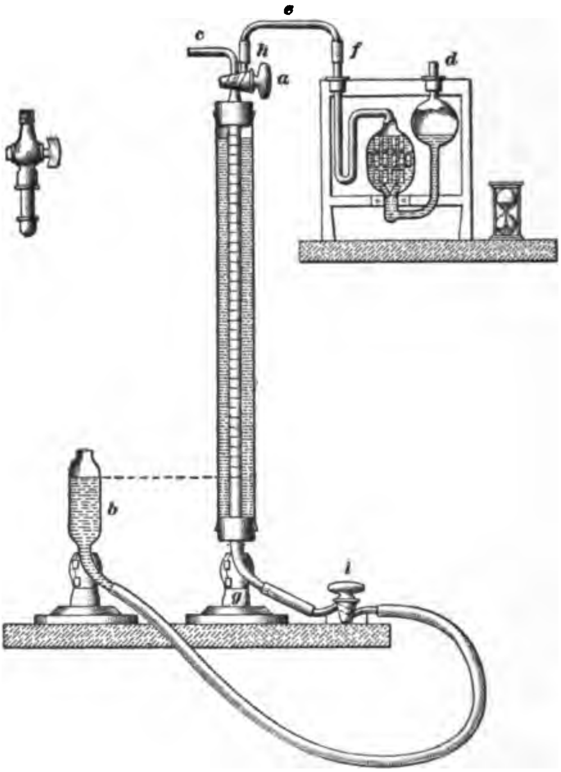

FIG. 4

to $\frac{2}{10}$ centimeter. A large glass tube, stopped at each end with rubber plugs, is placed outside the burette. The space between the burette and the outside is filled with water, forming a water-jacket, which maintains the gas at an even temperature when in the burette.

A leveling bulb is connected, by a long rubber tube, with the bottom of the burette. This bulb is filled with water, preferably distilled, which has been saturated with gas by allowing a small stream of gas to bubble through it.

28. Operation of the Gas-Testing Apparatus.—Open lower stop-cock *l*, and turn three-way stop-cock *a* so that burette is open to pipette; then, by lowering level bulb *b*, draw the potash solution up capillary tube to the point *e*, just before capillary turns down, and close stop-cock *a*. Leaving lower stop-cock open, turn stop-cock *a* until capillary *c* is open to burette, and then, by raising level bulb *b*, fill burette completely full of water. Close stop-cock *l*. Now attach rubber tube to gas supply, and allow gas to flow through the tube for a moment to displace air; then, with gas still flowing, attach free end of tube to capillary *c*. Open lower stop-cock *l* and draw in gas to the mark 100 c. c. Close stop-cock *l*, and then close stop-cock *a* and detach rubber tube. After 3 minutes bring the level of the liquid in the burette exactly to 100 c. c. merely by raising or lowering level bulb, and close lower stop-cock *l*. Open stop-cock *a* to capillary *c* for a moment, in order to allow surplus gas to escape. There will be exactly 100 c. c. of gas in burette, measured under atmospheric pressure. Now open stop-cock *a* to pipette and force gas over to pipette by raising level bulb; draw gas back into burette immediately, letting potash solution follow up capillary to point *e*, as before, and close stop-cock *a*. After 3 minutes, by raising or lowering level bulb *b*, bring the water in burette and level tube to the same level and close stop-cock *l*. Note the point at which the water now stands in the burette, and the difference between this reading and the original amount taken will be the carbonic-acid gas absorbed.

The glass stop-cocks of the apparatus should be kept greased, as otherwise they may stick and break when an attempt is made to turn them. A glass cock that has stuck can usually be loosened by the application of a cloth wet with hot water.

In order to prevent the absorption of carbonic acid from the atmosphere, the open ends of both level bulb and pipette should be plugged when the apparatus is not in use.

Where unpurified gas is to be tested, the sulphureted hydrogen should be removed by passing the gas through a small oxide purifier before it is drawn into the burette. The solution of caustic potash in the pipette will last 5 or 6 months before it must be removed.

This method of absorption is the base of all the usual forms of gas analysis, different chemicals being used to replace the caustic potash and absorb the different components of the gas to be analyzed.

COKE OVENS

29. Modern Coke Ovens.—A new departure in gas manufacture is the **by-product coke oven.** For years there had been an annual waste of millions of feet of gas and of thousands of dollars' worth of tar and ammonia in the process of making coke in the old beehive ovens. There are two general styles of coke ovens that save or make use of all the by-products and produce good metallurgical coke. These are the *Otto-Hoffman* and *Lowe* types.

30. The Otto-Hoffman Ovens.—The Otto-Hoffman ovens are built in groups, each individual oven retort being about 30 feet long by 6 feet high by 18 inches wide. There are flue spaces between the ovens. The ovens are heated by regenerative furnaces of the Siemens type. The air supplied to the furnaces is forced by a blower through a checker work of heated brick, and is then admitted to the combustion chambers below the ovens, where it meets a supply of gas and combustion takes place. The hot gaseous products of combustion pass up through the vertical flues between the ovens and then down through another set of flues, through another checkerwork, and out to the stack.

When the checker brick, which are heating the incoming air, begin to get cold, the operation is reversed by a special valve, so that the air goes in through the checker-brick chamber, which has been heated by the hot flue gases passing to the stack, and the flue gases are passed through the checker brick that have cooled, heating them up again. The air blast is usually reversed every half hour.

31. There are two sets of mains, corresponding to the hydraulic main in a coal-gas plant, over the ovens, and the gas made during the first half of the time a charge of coal is being carbonized is conducted through one set of mains and condensed, scrubbed, and purified, similar to ordinary coal gas, and stored for domestic use. The gas made during the last half of the time the charge of coal is being carbonized is burned in the regenerative furnace. No other fuel is used to heat the ovens. It is a well-known fact that the gas produced during the first part of the time a charge of coal is being carbonized is much richer than that made during the last half, and on this account the commercial gas produced by this process is of excellent quality.

32. The ovens are charged by dumping in the coal through three coaling doors on top. A track is provided along the top of the setting, so that the coal can be dumped in from a car. After being charged, the coal is leveled by a long rake and the charge is allowed to remain in the retort for about 36 hours. About 6 tons of coal are put in at one charge. When the coke is ready to be removed, a door at each end of the oven is opened and the entire charge of coke is pushed out bodily by a ram, or pushed into an iron car, where it is quenched with water.

It is then dumped directly into freight cars that stand on a track running parallel to the open setting. The coke produced by this process is equal to the best oven coke for metallurgical purposes, and the yield of tar and ammonia is about double that usually obtained from the same weight of coal. An inferior coal or slack, which would not give satisfaction in gasworks, gives excellent results. The ovens are usually built in groups of fifty, and owing to the introduction of machinery for almost every operation, the expense for labor is far less than in gasworks handling the same amount of coal.

33. Where a richer gas is desired than that produced during the first half of a charge, the gas from the second half of the charge may be scrubbed with dead oil before it is burned, and the illuminants thus removed may be added to the commercial gas. About 4,000 cubic feet of commercial gas is produced per ton of coal. The installation, however, is very costly, and a good market for coke is necessary to make the process a financial success.

34. The Lowe Coke Ovens.—The Lowe coke ovens work nearly on the principle of the old-style beehive ovens. The ovens are about 30 feet long by 7 feet high by 5 feet wide. They are set side by side in groups of six or eight, and at each end of the setting is built a superheater filled with checker brick in the lower part and iron bars in the upper part. There is also a stack at each end. There are no combustion flues, but the ovens all communicate with one another near the top and with the superheater and stack at each end. There is a stack valve at each end similar to that used in an ordinary water-gas plant, and also a gas take-off pipe with a seal box similar to that used in a water-gas plant, also the proper scrubber and condenser.

35. The ovens after being heated to the proper temperature are charged with several tons of coal through charging doors on top of the setting. Dumping cars are provided, as with the Otto-Hoffman ovens. The charges are leveled and the stack damper is opened at one end; air is admitted at the other end and at one or more intermediate points. The air is heated as it comes through the checker brick at the end, and by the flues leading in at the intermediate points. Combustion takes place above the coal in the ovens, where the gases that are driven out by the heat meet the hot air, and thus the heat is kept up. When sufficient time has elapsed to get the required heat in the ovens, the air supply is shut off and the stack valve is closed. Less than one-half the gas is required for heating. The gas is then taken off through the take-off pipe at the end where the stack valve has been opened. Water is allowed to trickle in on the hot-iron bars at the other end and is turned to steam. This steam is raised to a high temperature in passing through the checker brick and combines with the free carbon, usually deposited as retort carbon, and a larger part of that deposited as tar turning it into gas. What little tar is produced is pumped back into the ovens and turns to either coke or gas. The coal is coked by the direct heat of combustion without the loss due to passing through retort or oven walls, and a very high yield of the best metallurgical coke results. The yield of ammonia is not so large as with the Otto-Hoffman ovens, but about 1,400 cubic feet of gas is produced per ton of coal.

When the heats begin to drop, the stack valve at the other end is opened and air is admitted at the end where the stack valve was open during the previous run. The superheater at this end has been heated by the outward passage of the hot gases and now seems in turn to heat the air and steam that is admitted. The superheater at the other end is meanwhile being again heated.

When the proper time has elapsed, the valves are again closed and gas taken off at the opposite end from where it was taken off before.

36. This gas is of low candlepower, but contains about 500 heat units and is excellent for gas engines and other fuel purposes. It can be readily brought to a candlepower suitable for ordinary city use by the addition of about 2 gallons of oil per thousand feet of gas made. The oil is sprayed in at the top of the ovens. Heavy crude oils, such as the California and Texas oils, are suitable for this purpose. The charges are burned off in about 24 hours, and the ends of the ovens are opened and the coke is pushed out by a pusher or ram, just as in the Otto-Hoffman process. Almost any grade of soft coal will give good results in these ovens.

The labor economy due to the use of machinery in all operations is fully as great in the Lowe plant as in the Otto-Hoffman plant.

It is necessary to use an exhauster with both styles of coke ovens.

ACETYLENE GAS

37. Acetylene is a pure hydrocarbon gas. It is represented by the chemical formula C_2H_2 (see *Inorganic Chemistry* and *Organic Chemistry*). Acetylene contains a higher percentage of carbon than any other hydrocarbon, the composition by weight being 92.3 per cent. carbon and 7.7 per cent. hydrogen. The gas is colorless and the commercial article has a strong odor suggestive of garlic. This odor is mainly due to the presence of small quantities of various impurities, and where these are not present there is only a slight and by no means disagreeable ethereal smell. Acetylene is readily soluble in water, at 15° C. absorbing its own volume of gas. At ordinary temperature 12 cubic feet of water will absorb about 13 cubic feet of acetylene. The specific gravity is .91, air being taken as unity. In burning 1 cubic foot of acetylene, 1,685 heat units are developed.

38. The light produced by burning acetylene diffuses better than any other light. It is a clear white light, very similar to sunlight, and all substances show their true colors when illuminated by it. The candlepower under the most favorable conditions is about 240 for a consumption of 5 cubic feet per hour, but it must be noted that it is impossible to get satisfactory or economical results with a flame giving more than 50 candlepower. Acetylene is usually burned through ½-foot burners, each burner yielding in ordinary practice 20 candlepower. Where more light is desired in one place, several such burners are grouped together.

MANUFACTURE OF ACETYLENE

39. Calcium Carbide.—Acetylene is produced commercially by adding water to a substance known as **calcium carbide.** Calcium carbide is a stone-like material, usually dark brown or black in color. It cannot be burned and will keep indefinitely if kept sealed from the air. If left exposed, the moisture in the atmosphere gradually slakes it and it eventually turns to slaked lime. It always gives off an acetylene odor, due to the fact that the moisture in the air is constantly decomposing it and producing small quantities of acetylene. Calcium carbide is produced by fusing together carbon, in the form of ground coke, and lime in the intense heat of an electric furnace.

When water is added to calcium carbide it is decomposed according to the following formula: CaC_2 (carbide) $+ 2H_2O$ (water) $= Ca(OH_2)$ (slaked lime) $+ C_2H_2$ (acetylene). One pound of absolutely pure calcium carbide will produce about 5.5 cubic feet of acetylene; but carbide as usually sold is not pure, and the yield of acetylene will be 5 cubic feet per pound or less.

40. Impurities.—The impurities found in calcium carbide, and consequently in acetylene, are phosphorus, sulphur, ammonia, and traces of hydrogen and silica. Any unfused carbon and the ash from the coke, that may be present in the calcium carbide, will be found in the residue of slaked lime after the gas has been driven off.

The phosphorus and sulphur, in the form of hydrogen phosphide and hydrogen sulphide, are the only impurities that might prove objectionable, and where good carbide is used, the quantities of these are so small that they may be neglected. Acetylene burns with almost perfect combustion, and no smell is noticeable from the burners. Where an odor of acetylene is discernible, it is an evidence that there is a leak.

In the production of acetylene a large amount of heat is generated. This heat may be localized so as to be harmful to the acetylene produced or it may be diffused so as to do no harm, according to the way the water and acetylene are brought together.

Thus, if water be allowed to drip slowly on a mass of carbide, local overheating will occur and the acetylene will be partly broken up into other hydrocarbon compounds of an oily nature; the candlepower will be much reduced, and there will be a tendency for the burners to clog and carbonize. Besides this, dry carbide when heated has a tendency to give off lime dust, which will choke the burners and may even fill up the pipes completely with a deposit of lime. This trouble may be partially overcome by the use of filters of cotton or thin cloth, or by making the acetylene bubble through a washer. Where the carbide is dropped in small lumps into a considerable volume of water, the water absorbs the heat, and the gas, cool and free from dust, bubbles out of the water.

The sludge of lime waste that is left after the acetylene has been all evolved should be of a whitish color, and if yellow or brown, it is a sure indication that the heats have been too high. This residue is harmless and will not burn. It packs well and makes good walks or drives, and as it consists mostly of the hydrate and carbonate of calcium, it is valuable as a fertilizer.

41. Dangerous Properties.—Acetylene is an endothermic compound. This means that heat is absorbed when it is developed and is given off when it is decomposed. If acetylene is compressed, so that the particles of gas are forced nearer together, by a pressure of 150 pounds to the square inch or more, it may be decomposed when subjected to high heats and may become violently explosive. The use or possession of liquid acetylene or of acetylene at high pressure is dangerous and should be avoided, but acetylene at ordinary temperature cannot be exploded unless it is mixed with air and ignited. If subjected to high heats at ordinary pressure without the presence of air, it will merely decompose into other hydrocarbon forms, such as benzene, methane, etc.

42. Acetylene, in common with all other combustible gases, is explosive when mixed with air in certain proportions. One part of acetylene mixed with 12.5 parts of air will produce perfect combustion and most violent explosion.

Moreover, the igniting temperature of acetylene is comparatively low. While ordinary coal gas ignites at about 1,100° F., acetylene ignites at about 900° F. It may therefore be lighted by a cigar or cigarette. It will be seen from the foregoing statement that acetylene must be handled with care; but in a mixture of 1 part in 10,000 parts air, it may be readily detected by the smell, and leaks may be located and stopped long before there is the slightest danger of any explosion.

It is dangerous, however, to look for leaks with a match or candle, and all tests should be made with a little soap and water. The soap suds should be brushed on wherever a leak is suspected, and the formation of soap bubbles will at once indicate its location.

With properly installed piping and a properly constructed and placed generator, there is absolutely no danger in the production of acetylene in the cellars or other suitable parts of residences, provided the apparatus is handled by a person who has been properly instructed, and provided good judgment and common sense are used. This is made evident by the fact that the Fire Underwriters make no objection to the installation of any one of the numerous generators that have been tested and approved by their experts.

GENERATORS

43. Types of Generators.—There are five general types of generators in use. These are the *spray*, the *overflow*, the *rescission*, the *dip*, and the *drop*, or *plunge*, types.

44. The Spray Generator.—The spray generator consists of a drum or shell, either cylindrical or square, into which a pan of carbide is introduced. Water is allowed to drip on to this carbide, the supply being usually cut off automatically by the increase in pressure or by the raising of the bell of a small gas holder, when gas is produced more rapidly than needed. The spray generators are sure to overheat the gas, producing the attendant burner trouble and loss of candlepower.

Steam is frequently generated when the water strikes the carbide, and lime dust, which clogs the pipes and burners, is apt to be thrown off. This type of generator is divided into two subdivisions, the dry and the wet. In the first form only enough water is added to produce the gas, the residue being removed in a dry state. These machines are wasteful because some of the carbide is frequently removed unused, and the unused carbide is sure to give the residue a very strong odor. In the wet machines enough water is run in to flood the carbide before it is removed.

On account of the overheating and the lime-dust trouble, the spray type of generator cannot be recommended except in certain extraordinary conditions, such as in bicycle lamps, where the spray, or drip, system is the only practical way to apply the water to the carbide.

45. The Overflow Generator.—In the overflow generator the carbide pan is divided into a number of small compartments so arranged that, when water is turned on the first compartment, it floods the carbide in that before it overflows and begins the generation of gas in the second. The compartments are thus successively filled and overflowed, until the carbide has been all flooded. The water feed may be automatically regulated, as in the case of the spray machine. Generators of this type are open to the same objections as the spray type, though perhaps to a lesser degree. Some of them give very fair results in practical use.

46. The Rescission Generator.—In the rescission generator the carbide is held suspended in baskets or perforated pans, and water is allowed to rise in the generator until it reaches the carbide and the production of acetylene begins. Should gas be produced faster than is needed, the pressure forces the water down away from the carbide and the generation ceases. Where the water comes up to the carbide and produces vigorous generation and then quickly recedes, the carbide is apt to become so hot as to be incandescent and great damage to the quality of gas results. Different generators have a greater or less tendency, as the case may be, toward this trouble.

47. The Dip Generator.—In this generator the carbide is placed in baskets or perforated pans that are suspended from the inside of the bell of a small gas holder or gasometer. As gas is used from the holder, it gradually lowers until the carbide comes in contact with the water in the tanks. Acetylene is then generated, and if the rate exceeds the demand, the gas accumulates under the bell and raises it until the carbide does not touch the water, when the generation of gas soon stops. The carbide is very apt to become overheated, just as in the rescission apparatus.

48. The Drop, or Plunger, Generator.—In this kind of generator the carbide is allowed to fall, a small piece at a time, into a comparatively large volume of water. The small piece of carbide being dropped into a large quantity of water, the water absorbs the heat as fast as it is generated, so that the gas is produced cool, that is, below 212° F., and is consequently of excellent quality. As the gas has to bubble up through the water, it is thoroughly washed and a large proportion of the impurities removed. The lime sludge drops to the bottom and is removed from time to time. It has been claimed that a considerable loss of acetylene resulted in this form of apparatus on account of the fact, heretofore stated, that 1 cubic foot of water will absorb a little over 1 cubic foot of acetylene. This is true if the gas has access to pure water, but as the water in the generator is constantly taking up lime, the gas absorbed when water is freshly added is largely driven out and the acetylene loss does not amount to much. Even if the water in the generator remained pure and 11 parts of gas were absorbed to every 10 parts of water, the loss would not be 3 per cent.

A 25-pound generator, containing 25 gallons of water, would in this case absorb about 27½ gallons of acetylene. As 1 cubic foot is equal to 7½ gallons, the loss would be 3.67 cubic feet. Since the amount of gas produced from 25 pounds of carbide would be about 125 cubic feet, the loss is found to be 2.9 per cent.

In theory, the amount of water required to produce all the acetylene from 1 pound of carbide is .56 pound, or a little over ½ pint, but 1 gallon to the pound is nearer the usual practice.

49. Storage Apparatus.—All generators should be provided with a storage tank of the gas-holder type, to take up and store the gas that generates after they have been turned off; otherwise this after generation may cause an undue pressure on the generators.

The gas-holder type is recognized as the best form of storage tank, because it serves both as a storage and as a reducing valve, maintaining a constant pressure in the piping system.

50. There is another form of storage apparatus that is sometimes used. This consists of two tanks that are connected together at the bottom and partly full of water, the top of one being open to the atmosphere. A large **U** gauge is practically formed, and when gas is admitted above the water in one tank, the pressure forces the water down in that tank and correspondingly up in the other. This process has the disadvantage of storing the gas at a pressure that is increased as gas is added, and some form of a reducing valve is therefore necessary to insure steadiness in the lights.

The granulated carbide is fed into the drip-type generator either by hand or automatically. The former method is suitable for large establishments and public lighting plants, where considerable holder capacity is at hand and where an attendant can give regular attention to the feeding. The latter is suitable for household purposes.

51. In the early history of acetylene lighting great difficulty was encountered in constructing an apparatus that would successfully feed granulated carbide automatically, but the problem has now been solved by many makers of generators.

52. All generators should be provided with a safety valve or seal opening at a pressure of not over 3 pounds, and connected with a pipe leading outside of the building. As no mechanical blow-off valve will work properly at the low pressures usually employed, the best blow-off is formed by a piece of pipe dipping 6 inches into water seal. Such a seal will blow off at 6 inches water pressure and will reseal itself when the pressure goes down, which a **U** gauge cannot do.

It should also be possible to charge a generator and to remove the residue without the escape of gas and without the admittance of much air to the inside of the generator.

Generators should never be connected with a greater number of lights than they are designed to carry, and they should preferably be operated on a smaller number of lights than which they are rated. Overheating is caused by overloading, and consequent too rapid generation, and is accompanied by its sure result, polymerization, or the breaking up of the acetylene in other hydrocarbons, such as benzene, anthracene, styrolene, etc.

53. Points of Excellence.—The following were the points on which judgment was passed in awarding medals to acetylene generators at the Pan-American Exposition:

1. The contact of the carbide should be with the water; or, in other words, the machine should be of the drop type.

2. The carbide should be fed into the water in proportion to the consumption of gas.

3. The gas pressure at the point of delivery should remain practically constant, irrespective of the number of jets burning, or of carbide, or of gas in the generator.

4. The pressure should be equal in all parts of the machine and should not exceed a 6-inch column of water.

5. When the lights are turned out, the generation of gas should cease.

6. The gas should be delivered to the burners clean, cool, and dry.

7. The heat of generation must not exceed 200° F.

8. When the generator is recharged, there should be no escape of gas.

9. If the generator is left idle for a long period, there should be no deterioration of the carbide.

10. The gas holder attached to the generator should be of ample capacity and made tight with a water seal.

11. The pressure in the service pipe should never exceed that of a 3-inch column of water, and a provision must be made to blow off in the air at a pressure of 6 inches.

12. The water capacity of the generator must be at least 1 gallon of water to 1 pound of carbide.

13. There must be a convenient method of getting rid of the slaked carbide without escape of gas.

14. The generator must be entirely automatic in its action; that is to say, after it has been charged it will need no further attention until the carbide is entirely exhausted.

15. There should be a simple method of determining the amount of unconsumed carbide in the generator at any time.

16. The generator should be so simple in operation that it can be tended by unskilled labor without danger of accident.

17. The various operations of discharging the refuse, filling with fresh water, putting in carbide, and starting the

generator should be so arranged that it is impossible to do them in the wrong order.

18. The generator must be so arranged that there can be no possibility of mixing air with acetylene gas.

19. Generators must be built of substantial materials well adapted to their purpose.

20. Generators must be so constructed that an addition to the charge of carbide can be made at any time without affecting the light.

Any generator that is built according to the preceding rules may be considered excellent.

54. Generators should not be made of tin, but of galvanized sheet iron not less than No. 20 gauge, but No. 22 gauge may be used in bell constructions, where stiffening rings are provided. Seams should be either locked or riveted, as well as soldered. Seams that are only soldered are liable to open up if a fire occurs external to the apparatus, and this in the case of an automatic-feed generator might result in the lowering of the bell through leakage, and a consequent large production of gas at a time when it would produce most harmful results.

Braces, pipe hangers, etc. should not be secured by bolts, but rather by rivets, and the heads of these should be soldered over to avoid any chance of a leak. All pipe connections should be of standard galvanized-iron pipe. Where pipes run through the sheet metal, either a flange, with a standard pipe thread tapped into it, should be riveted or soldered on, or malleable locknuts should be soldered on each side of the sheet metal. Pipes should run through sheet metal at right angles to it.

Right-and-left couplings properly made up are better than flange unions or packed unions, ground unions, or long screws. Where flanges are necessary, the best pure rubber packing not less than ⅛ inch thick should be used.

The pipe connections should be as simple as possible, and the minimum number of valves and fittings should be used.

55. The height of the gas holder should be about the same as its diameter, and the size should be such that when no gas is being generated and the machine is feeding the maximum number of lights for which it is rated, the bell will descend at the rate of not more than 3 inches per minute. The bell should be guided either by a central guide post or by side columns on the same principle as the ordinary large gas holder. Just before the bell can reach its maximum height an automatic blow-off should open into the waste pipe that runs to the outside of the building. In this way all danger of the gas holder getting too full and allowing gas to blow out into the room will be avoided. The inlet and outlet pipes should be separate and placed as far apart as possible. Variation of pressure in the house pipes, due to the rapid production of gas in the generator, will thus be avoided, and the gas will have a better chance to cool and deposit some of its moisture.

The space in a machine that will contain air when starting up should be made as small as possible.

No copper should be used in any part of the construction of an acetylene machine.

In the presence of some of the impurities found in acetylene, a substance known as acetylide of copper is formed. This substance belongs to that class of material known as fulminates, and in common with them it can be exploded by a sharp blow or by friction.

56. Safety Regulations.—The following regulations should be observed in the operation of acetylene machines:

1. Calcium carbide should be kept in water-tight metal cans by itself, outside any insured building, under lock and key, and where it is not exposed to the weather.

2. A regular time should be set for attending to and charging the apparatus during daylight hours only.

3. In charging the generating chambers of water-feed machines, clean all residuum carefully from the containers and remove it at once from the building. Separate from

the mass any unslaked carbide remaining, and return it to the container, adding new carbide as required. Be careful never to fill the container over the specified mark, as it is important to allow for the swelling of the carbide when it comes in contact with water. The proper action and economy of the machine are dependent on the arrangement and amount of carbide placed in the generator. Carefully guard against the escape of gas.

4. Whenever charging with carbide, always replenish the water supply.

5. Never deposit residuum or exhausted material from water-feed machines in sewer pipes or near inflammable material.

6. Water tanks and water seals must always be kept filled with clean water.

7. Never install more than the equivalent of the number of ½-foot burners for which the machine is rated.

8. *Never test the generator or piping for leaks with a flame, and never apply flame to an outlet from which the burner has been removed.*

9. *Never use a lighted match, candle, lantern, or any open light near the machine.*

10. See that the entire installation is in accordance with the rules of the National Board of Fire Underwriters, a copy of which will be furnished by your insurance agent, and obtain from your contractor a written guarantee that these rules are complied with.

NOTE.—Failure to observe the foregoing cautions is liable to endanger life as well as property.

57. Installing Generators.—Since water is used in acetylene generators and gas holders, they should be installed in places where the temperature will not go below the freezing point. Where this is impossible, the water drawn from the generator with the residue should be allowed to stand in a tank or barrel until the lime has settled out of it. The clear water may then be put back in the machine and will not freeze above 0° F. Ten per cent. of glycerine added to the water in a gas holder will prevent freezing under ordinary conditions. Stoves and fireplaces must never be permitted in the same room with the generator. The generator room may be readily heated, where hot water or steam heat is not at hand, by putting in a coil of hot-water pipe, which may be supplied with hot water from a waterback in a range or stove situated in another room.

58. Calcium carbide costs about 4 cents per pound delivered in small quantities on the consumers' premises. Since 1 pound of calcium carbide will produce at least 4½ cubic feet of acetylene, the cost may be reckoned at a little less than 1 cent per foot. The cost therefore for each ordinary ½-foot burner will be about ½ cent per hour for 20 candlepower.

Ordinary city gas at $1.50 per thousand cubic feet costs ¾ cent for each open flame burning 5 feet per hour. City gas is usually about 20 candlepower, so that it would require one 5-foot burner to give as much light as one ½-foot acetylene burner. When used with Welsbach lights, however, city gas gives about 20 candlepower per cubic foot, so that a light burning the usual quantity of gas about 3½ cubic feet per hour would produce about 70 candlepower at a cost of .525 cent per hour.

59. Acetylene Burners.—Ordinary gas tips cannot be used with acetylene. This is because the acetylene flame from an ordinary gas tip does not get sufficient air for perfect combustion and consequently smokes; moreover, the tip soon becomes so hot that the acetylene is polymerized or decomposed by the heat before it is burned, and the candlepower of the flame is lowered, while the tip itself soon becomes choked with soot and carbon, which are produced by the charging of some of the more condensable hydrocarbons produced in the polymerization.

The best acetylene burners are made in the form of a **Y**. The small jets, one from each branch of the **Y**, impinge upon each other and form a flat flame at right angles to the plane in which the jets stand. Each jet is a miniature Bunsen burner that draws air along with the gas so that just enough air is supplied to make smokeless combustion. The flame itself does not touch the tips, which consequently do not become overheated. Metal burners are apt to warp, even at the low temperature to which they are subjected; the two jets may be so deflected as not to impinge properly, and therefore steatite burners are best in most cases.

It is bad policy to turn down ordinary acetylene burners, as the jets then lose their Bunsen effect and are apt to become clogged with soot. There are, however, burners that are supplied with a small needle valve regulating each jet, and they may be regulated to burn a very small amount of acetylene with satisfactory results.

The use of acetylene in lamps of the Welsbach type has been frequently attempted and there are several such lamps on the market. The highest practical efficiency obtained by the use of acetylene with Welsbach lamps is about 90 candlepower to the cubic foot, and a ½-foot burner of this type would therefore give but 45 candlepower, which is little more than double the light that can be obtained by the open flame.

It has been found that the intense heat is very trying to mantles, and if the use of Welsbach lights is to be made successful, it is necessary to remove every trace of phosphureted hydrogen from the acetylene; otherwise, phosphates of the earth of which mantles are composed are soon formed on the surface of the mantle, and as these phosphates are readily fusible, the life of the mantle is short.

PHOTOMETRY

60. The capacity of the human eye for the perception of light is comparatively small. It is unable to perceive very faint lights, and it is dazzled and confused by lights of great brilliancy. Photographic plates are affected by faint lights that are invisible to the eye; thus, photographs of the sky reveal a multitude of stars that are not visible even with the aid of the strongest telescopes. The unaided eye is unable to judge of the relative intensity of various lights with any reasonable approach to accuracy. The art of measuring the comparative intensity of lights is called **photometry.** There are several methods of making these measurements—chemical, electrical, and mechanical—each of which is peculiarly suited to special cases. The method employed for general purposes is to compare the illuminating power of the light under examination with that of a light of standard intensity.

The unit that is used for all ordinary measurements is the light given by a sperm candle that burns at the rate of 120 grains per hour.

The candle is burned in still air, and care is taken to avoid all drafts that might accelerate the combustion and thus vary the brilliancy of the light. The light thus obtained is made the unit for comparison, and is called **1 candlepower.**

PHOTOMETERS

61. All instruments that serve to measure the comparative brilliancy of lights are properly called **photometers,** but only those that are suitable for measuring ordinary gas lights, etc. will be described.

62. Rumford Photometer.—One of the oldest of these instruments, called the **Rumford photometer,** is shown in Fig. 5. It consists of a table having a black wooden post *c*, standing erect, and a screen *g*, which receives the shadows of the post that are cast by the lights *a* and *b*. The candle *a* is the standard light, and *b* is the light whose intensity is to be measured. The lines *h c* and *i c* make exactly equal angles with the screen, and the lights are moved back and forth along these lines until the shadows *e* and *f* appear of exactly equal blackness. The powers of the two lights are then computed by dividing the square of the distance *b c* by the square of the distance *ac*, the quotient being the candlepower of the light *b*. This method is very inaccurate, and is not to be recommended, because the eye is unable to compare the shadows *e* and *f* with the requisite accuracy.

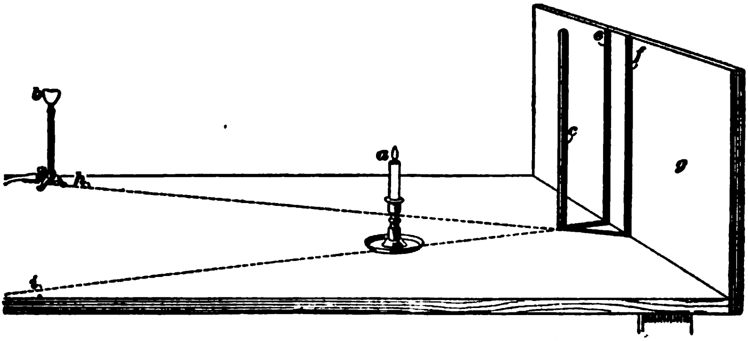

FIG. 5

63. Bunsen Photometer.—The **Bunsen photometer,** shown in Fig. 6, operates on a different principle. A diaphragm *c* is illuminated on its opposite sides by the light *b* and the standard candle *a*. The observer looks down through the tube *e* into mirrors *f* and *g*, and thus sees the reflection of both sides of the diaphragm at the same time. If they appear of unequal brilliancy, the sight-box *d* is moved along the bar *h* until they become equal. The candlepower of the light *b* is then found by dividing the

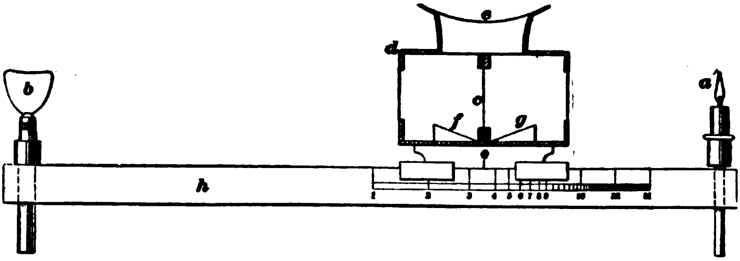

FIG. 6

square of the distance *bc* by the square of the distance *ac*. Usually the bar is graduated, as shown, so that no calculation is necessary.

64. Spot and Star Diaphragms.—There are two methods in vogue of constructing the diaphragm. The **spot diaphragm** is shown in Fig. 7. The center *a* is a disk of opaque white paper. The ring *b* is made of white paper that is saturated with paraffin and is translucent. The outer part *c* is blackened. When this diaphragm is unequally illuminated on its opposite sides, the ring *b* looks darker or brighter than the center *a*; but when the illumination is exactly equal, all difference disappears and the spot *a* becomes indistinguishable.

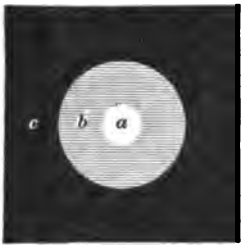

FIG. 7

The **star diaphragm** is shown in elevation at *A*, and in section at *B*, Fig. 8. It consists of a piece of white writing paper *a* of moderate thickness, having a star-shaped figure cut out of its center, and a sheet of thin white writing paper *c* of best quality, which is doubled so as to enclose the piece *a*. The diaphragm is lightly squeezed between two pieces of glass *b*, *b*. Care is taken in cutting the star to make every point and line clear and sharp. When the reflection of the diaphragm is seen in the mirrors, the images will vary in distinctness if the lights are unequal. The sight-box *d*, Fig. 6, is then moved along the bar until both images of the star appear equally sharp and clear.

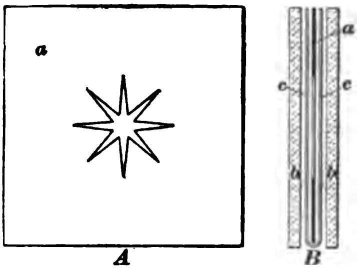

FIG. 8

65. It will be observed that the methods of testing employed in the photometers described are quite different. In the Rumford method the observer judges the equality in blackness of the shadows produced; in the Bunsen method, using the spot diaphragm, he judges by the equal brightness of the opposite sides of the diaphragm, and when using the star diaphragm, he judges by the equal clearness and distinctness of the two images of the star. The Rumford method has been discarded for the more accurate Bunsen method. Both the spot and star diaphragms are widely used; but the star diaphragm is preferred because of its superior accuracy.

66. In a third form of photometer the diaphragm is composed of a flat piece of chalk or other non-translucent white substance. Prisms are used to reflect the two sides of the diaphragm into the eyepiece of the sight-box, and these prisms are so arranged that a double rectangular field is produced. Each side of the field embraces a view of part of the reflection of each side of the diaphragm.

When the lights on each side of the diaphragm are exactly equal, the different parts of the field seen on looking through the eyepiece are indistinguishable; but if one side of the diaphragm be illuminated more strongly than the other, the parts of each field reflected from that side of the diaphragm stand out brighter than the other parts. This form of photometer is capable of even more accurate work than the spot or star type. It is largely used in Germany.

The sight-box is usually made reversible, so that by taking a number of observations with one side of the diaphragm toward *a* and then reversing the sight-box and taking the same number of observations with the other side of the diaphragm toward *a*, any difference in the two sides of the sight-box will be equalized.

67. Substitutes for Candles.—On account of the difficulty in obtaining candles of uniform make and the trouble of weighing them, several substitutes have been devised. Among these are the Hefner lamp, the Pentane lamp, the Standard Argand burner with Edgerton slit, and the Carcel lamp.

The Hefner lamp burns purely Amly acetate, the flame being about the shape of a candle flame and 40 millimeters high. The flame is fed by a wick in a tube 8 millimeters inside diameter. The light of the Hefner lamp is .88 candlepower. It is a very accurate and convenient standard.

The Pentane lamp is usually of 10 candlepower and burns "carburine," or pentane vapor, which is very light petroleum vapor. This lamp is also a very accurate standard and has been largely adopted.

The Standard Argand burner, with Edgerton slit, is a Sugg Argand burner provided with a black metal chimney in which a slit has been cut through which shines the light from a certain zone of the Argand flame. Ordinarily, gas is burned in this lamp, and its flame is regulated to a certain light. The light from this standard varies somewhat with the quality of the gas, but it is constant enough for everyday work where great accuracy is not required, provided the gas used in it is either straight coal gas or straight water gas. With varying mixtures of the two, it is unreliable. It should be tested frequently against standard candles or a Hefner or Pentane lamp. It gives about 5½ candlepower with coal gas.

The Carcel lamp is almost exclusively used in France. It is a simple wick lamp constructed to burn about 42 grams of colza oil per hour. It is not as accurate as either the Hefner or the Pentane lamp.

68. Calculation of Candlepower.—When candles are used, it is frequently the practice to cut them in two in the middle and after shaving off enough sperm to allow lighting the wicks, to burn the two ends at once, producing a light of 2 candlepower. The candle balance, used for weighing the candle as it burns, is frequently arranged to hold two candles. In practice, the distance between the centers of the lights on a photometer is usually either 60 inches, 100 inches, or 250 centimeters. In any case, the relative intensity of the lights at *a* and *b*, Fig. 6, may be estimated by dividing the square of the distance *bc* by the square of the distance *ac*. If the light at *a* is 1 candlepower, the candlepower of the light at *b* is at once obtained.

Example 1.—On a 100-inch photometer the sight-box is at a distance of 82 inches from *b* and 18 inches from *a*. The light at *a* is 1 candlepower. What is the candlepower of the light at *b* ?

Solution.— $82 \times 82 = 6{,}724$; $18 \times 18 = 324$; $6{,}724 + 324 = 20.75$ candlepower. Ans.

If the light at *a* be more or less than 1 candlepower, then the result obtained by dividing the square of the distance *b c* by the square of the distance *a c* must be multiplied by the candlepower of the light *a* to obtain the true candlepower of the light *b*.

Example 2.—On a 100-inch photometer the sight-box is at a distance of 67 inches from *b* and 33 inches from *a*. The light at *a* is 5 candlepower. What is the candlepower of the light at *c* ?

Solution.— $67 \times 67 = 4{,}489$; $33 \times 33 = 1{,}089$; $4{,}489 + 1{,}089 = 4.126$; $4.126 \times 5 = 20.63$ candlepower. Ans.

69. The scale on the bar is sometimes so arranged that if a light of 1 candlepower is used as *a*, the candlepower may be read directly from the scale by observing the figure at the point where the center of the sight-box stands when the lights on both sides of the diaphragm are equal. In this case, if the light at *a* is more or less than 1 candlepower, the figure observed on the scale must be multiplied by the value of the light to get the true candlepower.

Example.—The position of the sight-box is at 24 candlepower on the scale on the bar. The light at *a* is .88 candlepower. What is the candlepower of the light at *b* ?

Solution.— $24 \times .88 = 22$ candlepower. Ans.

70. An extremely accurate meter is always used to measure the amount of gas burned at *b*. This should be as nearly as possible at the rate of 5 cubic feet per hour in the case of ordinary illuminating gas. The temperature of the gas as it goes through the meter and also the height of the barometer should be observed, and the volume of gas registered by the meter should be corrected to 60° F. and 30 inches barometer.

The candlepower as figured from the scale on the bar must then be multiplied by 5 and the result divided by the corrected volume of gas registered to give the correct candlepower.

Example.—The uncorrected power of a gas is 22. The barometer is 30.3 and the thermometer stands at 54. The meter shows that gas is being used at the rate of 5.2 feet per hour. What is the corrected candlepower ?

Solution.—The corrections for barometer and temperature equals 1.025; $5.2 \times 1.025 = 5.33$; $22 \times 5 = 110$; $110 \div 5.33 = 20.64$ corrected candlepower. Ans.

The meters for photometer use usually have the dial geared up so that the rate per hour at which gas is passing the meter can be noted in observations of 1 minute in length.

71. Where a candle is used as a standard light, it should be lighted at least 15 minutes before the test is to commence. The candle must then be weighed and ten observations, five with the sight-box reversed, if this is possible, should be made on the photometer within the next 10 minutes. At the end of 10 minutes the candle must be again weighed, and if it is not burning at the rate of 120 grams an hour, the candlepower as corrected for temperature and barometer must be also corrected for the candle by multiplying it by the number of grams of sperm burned per hour and dividing the result by 120.

Example.—The candlepower of a gas corrected for barometer and temperature is 20.64. The candle was burning at the rate of 123 grams per hour. What is the true candlepower ?

Solution.— $20.64 \times 123 = 2{,}538.72$; $2{,}538.72 \div 120 = 21.16$ corrected candlepower. Ans.

72. While it is possible to make candlepower tests in a rough way with the aid of curtains and screens of black cloth, it is necessary to have a dark room or to work at night if reliable work is expected.

Gas of 18 candlepower and under should be burned in a Standard Argand burner. Gas of over 18 candlepower should be tested while burning at the rate of 5 cubic feet per hour through a 7 or 8 foot tip.

Jet photometers are frequently used in small gasworks. They are fairly accurate on gas for which they are made, but a jet photometer made for coal gas will not work properly on water gas, and vice versa.

INDEX

NOTE.—All items in this index refer first to the section and then to the page of the section. Thus, "Plaster of Paris, 46 32" means that plaster of Paris will be found on page 32 of section 46.

A	*Sec.*	*Page*
Abel closed-cup oil tester	53	11
Absorption apparatus for sulphur dioxide	47	39
“ of sulphur dioxide	47	39
Acetylene	55	22
“ burners	55	34
“ gas generators, Installing of	55	33
“ gas generators, Points of excellence of	55	29
“ machines, Safety regulations of	55	32
Acid treatment of lubricating oils	52	14
Action of gas holders	54	37
Agalite, Analysis of	48	28
Agitator	52	20
Alkali refining test of petroleum	53	20
“ treatment of lubricating oils	52	44
Alkalies	46	36
Alum, Analysis of	48	22
“ treatment of water	47	93
Ammonia stills	54	39
Ammoniacal liquor	54	38
“ liquor, Strength of	54	40
Analyses of Portland cements	45	37
Animal size for paper	47	75
Arrangement of furnace pipes	54	13
“ of sugar factory	50	11

B	*Sec.*	*Page*
Ball mill	45	15
Baltimore and Ohio railway specifications for oils	53	30
Barre and Bondel's nitric-acid process of wood pulp	47	52
Beating engine	47	67
“ engine, Jordan's	47	69
“ of fibers	47	67
Beds for topped sugar beets	50	7
Beet-sugar factory, Arrangement of	50	11
“ sugar making, Outlines of	50	14
Benzine and burning oils, Distillation test for	53	8
“ Test for oil of	53	8
Bisulphite liquor, Analysis of	43	45
“ liquor for the sulphite process	47	36
Black ash, Leaching of	47	34
Bleach, sludge, Analysis of	48	20
Bleached stock, Treatment of	47	65
Bleaching, Electrolytic	47	59
“ of esparto	47	56
“ of fibers	47	53
“ of ground wood	47	57
“ of jute	47	56
“ of manila	47	56
“ of rags	47	55
“ of straw	47	56
“ of sulphite pulp	47	58
“ of wood fiber	47	57
“ powder, Analysis of	48	19
“ powder, Preparation of solution	47	54
Boiler, Bertam's, for rags	47	7
Boiling-water test	46	18
Bone black, Decolorizing of	50	68
Breaking briquets	46	11
“ briquets, Speed of	46	13
Briquet-breaking machine, Fairbanks	46	12

Sec. Page
Briquet-breaking machine, Riehle's ... 46 11
Briquets, Breaking of ... 46 11
" for testing purposes 46 7
" Sand and cement ... 46 10
Broke beater ... 47 69
Brown size for paper ... 47 72
Burgess absorption apparatus for sulphur dioxide ... 47 40
Burning of lime ... 45 10
" of natural cements ... 45 20
" of Portland cement ... 45 50
" oils, Grading of, according to color ... 52 26

C Sec. Page
Calcination of Portland cement ... 45 56
Calcium carbide ... 55 23
" carbide, Dangerous properties of ... 55 24
" carbide, Impurities of 55 23
Caiculation of candlepower ... 55 40
" of cement mixtures ... 45 37
" of soda recovery ... 47 35
Calendering of paper ... 47 85
Calorization of sugar-beet juice ... 50 29
Cane juice, Composition of ... 49 5
" sugar, Curing of ... 49 45
Cannel coal ... 54 4
Carbon dioxide ... 46 35
Carbonation, Difficulties of ... 50 32
" of sugar-beet juice ... 50 30
Carbonic-acid gas, Determination of, in water gas ... 55 17
Cardboard or pasteboard ... 47 91
Casing the hole of oil wells ... 51 25
Causes of unsoundness of cement ... 46 18
Caustic liquor, Analysis of ... 48 48
Causticizing lime, Analysis of 48 12
Cellulose ... 47 2
" Chemical properties of ... 47 3
Cement, Coloring matter in ... 46 37
" Composition of compounds of importance in ... 45 7
" Constancy of volume of ... 46 17
" Determination of specific gravity of ... 46 21
" History of ... 45 1

Sec. Page
Cement, Magnesian ... 45 35
" manufacture, Relation of chemistry to ... 45 1
" mixtures, Calculation of ... 45 37
" Packing and shipping 46 49
" Points to be observed in testing ... 46 14
" Portland, Burning of 45 50
" Portland, Calcination of ... 45 56
" Portland, Chemical analysis of ... 46 28
" Portland, Dry rotary-kiln process of ... 45 48
" Portland, Final pulverizing of ... 45 60
" Portland, Fineness test of ... 46 1
" Portland, Fusion of ... 45 58
" Portland, Influence of heat on setting time of ... 46 7
" Portland, Nature and composition of ... 45 36
" Portland, Physical tests of ... 46 1
" Portland, Processes of manufacture of ... 45 42
" Portland, Setting time of ... 46 2
" Portland, Testing ... 46 1
" Portland, Use of gypsum in ... 46 33
" Rock ... 46 42
" Sampling of ... 46 38
" Seasoning of ... 46 37
" silica Portland, Manufacture of ... 45 31
" silica Portland, Properties of ... 45 31
" slag Portland, Manufacture of ... 45 29
" Specific gravity of ... 46 21
" Unsoundness of ... 46 18
" Uses of ... 46 43
" works, Location of ... 46 40
Cements, Classification of ... 45 17
" natural, Burning of 45 20
" natural, Composition of ... 45 8
" natural, Manufacture of ... 45 20
" natural, Pulverizing of ... 45 21
" natural, Tests on ... 45 24

Sec. Page

Cements, Portland, Analyses of 45 87
" Pozzuolana 45 25
" Quick-setting and slow-setting 46 3
" Slag 45 26
" slag, Composition of 45 28
" slag Portland, Properties of 45 30
" various, Tensile strength of 46 15
Center seal 54 29
Centrifugals 50 47
Chalk 46 43
Charcoal, Revivification of 50 67
Charging sulphite digesters 47 44
" the bench 54 19
Chemical analysis of Portland cement 46 28
" analysis of raw materials 46 27
" composition of petroleum 51 1
" constituents of sugar beets 50 10
" treatment of distillate for burning oils 52 18
" treatment of naphtha 52 28
Chemicals for paper analysis 48 4
Chilling of paraffin 52 37
Chipping of wood 47 15
Circular diffusion batteries 50 24
Clarification of sugar-cane juice by Howard's process 49 24
Classification of cements 45 17
" of limes 45 18
Clay 46 43
" Analysis of 48 30
Clogging of stand pipes 54 17
Cloud test for illuminating oils 53 16
Coal gas, By-products of 54 7
" gas, Composition of 54 6
" gas, Manufacture of 54 4
Coefficients, Definitions of 50 55
Coke filter 55 11
" ovens 55 18
Coking coal 54 4
" coals, Composition of 54 5
Color test for illuminating oils 53 16
" value of pigments 48 49
Coloring matter in cement 46 37
" of paper 47 77
Composition of cane juice 49 5
" of compounds of importance in cement 45 7
Composition of foreign hydraulic limes 45 16
" of natural gas 51 18
" of slag cements 45 28
" of slags 45 27
Compounded oils 52 51
Compressive strength 46 16
Concentration and granulation of sugar-cane juice 49 30
" of sugar-beet juice 50 41
Concrete 46 44
" Impermeable 46 47
" Mixing of 46 45
" mixtures 46 47
" Proportions of ingredients in 46 46
Condensers 54 24
" for petroleum 52 4
Constancy of volume of cement 46 17
Construction of rig for oil wells 51 21
Cotton 47 8
Cracking process 52 12
Crushing sugar cane 49 9
Crystallizable and uncrystallizable sugar 49 5
Crystallizers for sugar-beet juice 50 49
Cultivation of sugar beets 50 4
Cumulative resolution 54 2
Curing of cane sugar 49 45
Cutting of paper 47 89

D Sec. Page

Decolorization of sugar by charcoal 50 66
Decolorizing power of bone black 50 68
Defecation and clarification of sugar-cane juice by lime 49 21
" of sugar cane 49 20
" of sugar-cane juice by calcium bisulphite 49 26
" of sugar-cane juice by sulphurous acid and lime 49 27
Definition of sugar 49 1
Definitions of the coefficients 50 55
Demarara yellow crystals 49 42
Description of furnaces for the distillation of coal 54 9
Destructive distillation 54 2

Sec. Page

Determination of carbonic-acid gas, Apparatus for 55 15
" of specific gravity of cement 46 21
Difficulties in lime kilns 50 36
Diffusion battery, Circular 50 24
" Difficulties met in 50 28
" of sugar beets 50 23
" of sugar cane 49 19
" Process of 50 26
" Temperature of 50 27
" Water for 50 27
Digesters for wood pulp 47 16
" Sulphite 47 43
" Sulphite, Charging of 47 44
Digesting of wood for pulp 47 19
Dip generator 55 27
Disposition of waste sulphite liquor 47 49
Distillation of coal 54 8
" of crude petroleum for lubricating stocks 52 14
" of crude petroleum for naphtha, etc. 52 8
" of naphtha 52 29
" of oil residues 52 34
Dome kilns 45 51
Drier 45 43
Drilling tools for oil wells 51 23
Drop or plunger generator 55 27
Dry rotary-kiln process 45 48
Drying ovens 48 3
Dutch standards for sugar 50 72

E Sec. Page

Electrolytic bleaching 47 59
Elevators and conveying machinery 45 61
Elliott closed-cup flashing-point tester 53 6
Elution process of sugar-beet molasses 50 56
Esparto, Bleaching of 47 56
" grass 47 4
" pulp 47 11
" pulp, Washing of 47 11
Evaporators for sugar-beet juice 50 43
Exhauster 54 21

F Sec. Page

Faija's warm-water test 46 18
Fairbanks briquet-breaking machine 46 12
Fibers, Bleaching of 47 58
Filter presses 50 40
" pressing, Difficulties in 50 38
" pressing in sugar-beet sludge 50 38
Filtering of lubricating oils 52 45
Filters for lubricating oils 52 46
Filtration of water 47 92
Final pulverizing of Portland cement 45 60
Fineness test of Portland cement 46 1
Finishing the paper 47 90
Fire-retorts 54 15
Flashing point, Determination of, with Elliott closed cup 53 7
Foster automatic oil tester 53 8
Fourdrinier, or paper machine 47 79
Fractional distillation, Test of crude petroleum 51 10
Freezing mixtures 53 25
Fuel for kilns 45 55
Furnaces for the distillation of coal 54 9
Fusion of Portland cement 45 58

G Sec. Page

Galena oils 52 54
Gas coals, Composition of 54 5
" History of 54 1
" holders 54 36
Gasoline, Odor and acidity test of 53 2
" or benzine, Testing of 53 2
Generation of sulphurous acid 49 29
Glue, Testing of 48 40
Governors 54 38
Grading of burning oils, according to color 52 26
Graining sirup in vacuum pans 49 41
Granulation of sugar in vacuum pan 50 45
Greases 52 54
Griffin mill 45 48
" mills for pulverizing natural cements 45 21
Grinding of wood 47 13
Grit, or tube, mill 45 46
Ground plan of soda-pulp mill 47 27
" plan of sulphite pulp mill 47 49
" wood, Bleaching of 47 57
Gypsum 46 32
" Use of, in Portland cement 46 33

H *Sec. Page*

Harvesting of sugar beets..... 50 5
Headlight oil, Specifications and requirements of......... 58 29
History of cement.............. 45 1
" of paper................ 47 1
" of sugar................ 49 2
Hot-air test..................... 46 18
" tar scrubber............... 54 21
Hydraulic limes................ 45 15
" limes, Calcination of............. 45 16
" limes, foreign, Composition of... 45 16
" main................ 54 13
Hydrometers.................. 48 2

I *Sec. Page*

Illuminating oils, Cloud test for............ 58 16
" oils, Color test for............ 58 16
Impermeable concrete.......... 46 47
Importance of initial setting... 46 4
Indications of setting.......... 46 4
Indicators for paper analysis.. 48 10
Initial setting, Importance of.. 46 4
Instrument for determining flashing point................ 58 6

J *Sec. Page*

Jewell filter for water......... 47 95
Jordan's beating engine 47 69
Jute.......................... 47 4
" Bleaching of............... 47 56

K *Sec. Page*

Kerosene oil, Specifications and requirements of............. 58 29
Kilns, Dome.................. 45 51
" Fuel for.................. 45 55
" Rotary.................. 45 54
" Shaft.................... 45 50
" Shoefer.................. 45 52

L *Sec. Page*

Leaching of black ash.......... 47 34
Lime, Burning of.............. 45 10
" Complete analysis of.... 48 13
" kiln in sugar-beet factories................. 50 35
" kilns, Difficulties in...... 50 36
" Slaked.................. 45 12
Limes, Classification of......... 45 13
" Fat or rich............. 45 13
" Hydraulic............. 45 15
Limes hydraulic, Calcination of.................. 45 16
" hydraulic, foreign, Composition of...... 45 16
" Intermediate............ 45 14
Limestone...................... 46 44
" Occurrence and forms of........... 45 9
Linen......................... 47 3
Liquor for soda process of wood pulp................... 47 25
Loading of paper............... 47 76
Location of cement works..... 46 40
" of oil wells. 51 21
Low-grade lubricating oils.... 52 49
Lowe coke oven................ 55 20
Lubricating agitator........... 52 43
" oils, acid treatment of......... 52 44
" oils, alkali treatment of......... 52 44
" oils, Chilling point of.............. 58 24
" oils, Cleveland open-cup fire-tester........... 58 20
" oils, Cold-test..... 58 24
" oils, Evaporation test of........... 58 28
" oils, Filtering of.. 52 45
" oils, Filters for.... 52 46
" oils, Low-grade.. 52 49
" oils, Manufacture of.............. 52 39
" oils, Specific gravity of........... 58 20
" oils, Testing of ... 58 20
" oils, Viscosity tests of............... 58 21

M *Sec. Page*

Maceration of sugar cane...... 49 16
Machinery, Elevators and conveying......................... 45 61
Magnesia 46 34
" lime, Analysis of..... 48 16
Magnesian cement.......... ... 45 35
Maintaining of the seal. 54 14
Manila, Bleaching of........... 47 56
Manufacture of natural cements 45 20
" of pulp........... 47 6
" of sulphur dioxide............. 47 37
Marl 46 43
Materials used in the manufacture of paper................. 47 2

Sec. *Page*
McDougald absorption apparatus for sulphur dioxide 47 39
Measurement of sugar-beet juice 50 28
Michaelis boiling-water test 46 18
Mill, Ball 45 45
“ Griffin 45 48
“ Grit or tube 45 46
“ Whitehall raw-stone 45 48
Mills, Griffin, for pulverizing natural cements 45 21
“ rock-emery, for pulverizing natural cements 45 23
Mineral castor oil 52 53
“ oils as lubricants 52 50
“ seal oil, Specifications and requirements of 53 29
Mixing concrete 46 15
Molasses 49 44
“ and cane-sugar sirups 50 61
“ and sugars, Sampling of 50 70
“ residue or vinasse 50 63
“ Tempering of 49 44
Mouthpieces 54 12

N *Sec.* *Page*
Naphtha, Chemical treatment of 52 28
“ Distillation of 52 29
“ still 52 29
Natural cements, Burning of 45 20
“ cements, Composition of 45 18
“ cements, Manufacture of 45 20
“ cements, Pulverizing of 45 20
“ cements, Tests on 45 24
“ gas, Composition of 51 18
“ gas, Occurrence of 51 16
“ gas, Theories of origin of 51 19
“ gas, Uses of 51 16
Neutral oil 52 42
Nitrohydrochloric-acid process of wood pulp 47 52
Non-coking coal 54 4
“ coking coals, Composition of 54 6
Normal pat test 46 17

O *Sec.* *Page*
Ocher, Analysis of 48 32
Oil barrels 51 29
“ for water gas 55 18

Sec. *Page*
Oil residues, Distillation of 52 34
“ strata 51 26
“ tester, Abel closed-cup 53 11
“ tester, Foster automatic 53 8
“ Transportation of 51 29
“ wells, Casing the hole of 51 25
“ wells, Construction of rig for 51 21
“ wells, Drilling tools for 51 23
“ wells, Production of 51 21
“ wells, Torpedoing of 51 28
Opening and cleaning the retort 54 19
Osmose process of sugar-beet molasses 50 58
Otto-Hoffmann oven 55 18
Outlines of beet-sugar making 50 14
Overflow generator 55 26

P *Sec.* *Page*
Packing and shipping of cement 46 49
Paper analysis, Chemicals for 48 4
“ analysis, Indicators for 48 10
“ analysis, Standard solutions for 48 5
“ Animal size for 47 75
“ Brown size for 47 72
“ Calendering of 47 85
“ Coloring of 47 77
“ Cutting of 47 89
“ Finishing of 47 90
“ History of 47 1
“ Loading of 47 76
“ machine or Fourdrinier 47 79
“ made by hand process 47 78
“ made by machine 47 79
“ Materials used in the manufacture of 47 2
“ Rosin size for 47 71
“ Sizing of 47 71
“ Slitting and rewinding machine for 47 89
“ Supercalendering of 47 86
“ Testing 48 54
“ Tub sizing of 47 85
“ White size for 47 72
Paraffin and wax distillates, Treatment of 52 36
“ Chilling of 52 37
“ industry 52 33
“ lubricating oils 52 43
“ Pressing of 52 37
Pasteboard or cardboard 47 91
Petroleum 51 1
“ Alkali refining test of 53 20

Sec. Page
Petroleum, American hydrocarbons isolated from.............. 51 3
" Boiler stills for refining............ 52 2
" Chemical composition of........... 51 1
" Condensers of...... 52 4
" crude, Comparison of color and specific gravity...... 51 9
" crude, Distillation of, for lubricating stocks......... 52 14
" crude, Distillation of, for naphtha, etc................ 52 8
" crude, Fractional distillation test of................ 51 10
" crude, Production of................ 51 21
" crude, Technochemical examination of......... 51 8
" Determination of specific gravity of 53 1
" Elementary composition of....... 51 6
" Flashing point and fire-test of........ 53 4
" Products manufactured from....... 51 5
" products, Refining of............ 52 1
" Receiving tanks for 52 8
" Stills for refining... 52 1
" Storage tanks for.. 52 8
" Sulphuric-acid refining test of..... 53 19
" Testing of.......... 53 1
Photometer of Bunsen......... 55 37
" of Rumford........ 55 36
Photometers, Sources of light for.... .... 55 39
Photometry......... .... 55 35
Physical tests of Portland cement....... .... 46 1
Pictet and Brélaz's process of wood pulp.................... 47 52
Pigments, Color value of..... 48 49
Pipe lines....................... 51 30
Planting of sugar beets........ 50 4
Plaster of Paris....... .... 46 32
Portland cement, Analysis of 45 37
" cement, Burning of.. 45 50

Sec. Page
Portland cement, Calcination of.................. 45 56
" cement, Chemical analysis of......... 46 23
" cement, Dry rotary-kiln process........ 45 43
" cement, Final pulverizing of............ 45 60
" cement, Fineness test of.................. 46 1
" cement, Fusion of.... 45 58
" cement, Influence of heat on setting time of....... .... 46 7
" cement, Nature and composition of....... 45 36
" cement, Processes of manufacture of.... 45 42
" cement, Physical tests of.................. 46 1
" cement, Setting time of.................. 46 2
" cement, Testing of.. 46 1
" cement, Use of gypsum in............ 46 33
Pozzuolana cements............ 45 25
Preliminary straining of sugar-cane juice.................... 49 19
Preparation of barrels for refined oil........ 51 32
" of wood for the sulphite process 47 43
Pressing of paraffin........... 52 37
Pressure gauge................. 55 12
Primary and secondary settings.......... .... 46 5
Process for making paper by hand................ 47 78
" of diffusion 50 26
" of making paper by machine............ 47 79
Production of crude petroleum 51 21
Products manufactured from petroleum..................... 51 5
Properties of slag Portland cement.......................... 45 30
Proportional station meters... 54 34
" station meters, Testing of...... 54 35
Pulp, Digesting of wood for... 47 19
" Esparto.................. 47 11
" Esparto, Washing of..... 47 11
" from rags................. 47 6
" Manufacture of......... 47 6
" rags, Washing and breaking of.................. 47 9

Sec. Page
Pulp, Straw 47 12
" Wood 47 13
" Wood, by Barre and Bondel's nitric-acid process 47 52
" Wood, by nitrohydrochloric-acid process 47 52
" Wood, by Pictet and Brélaz's process 47 52
" Wood, by sulphate process 47 52
" wood, Liquor for soda process 47 25
" wood, Preparing of 47 18
" wood, Screens for washing of 47 21
" wood, Sulphite process 47 36
" wood, Washing of 47 19
Pulverizing of natural cements 45 21
Purification of water 47 92
Purifiers 54 27
Purifying material 54 29

Q Sec. Page
Quick-setting and slow-setting cements 46 3

R Sec. Page
Rag boiler 47 7
" engine 47 9
Rags, Bleaching of 47 55
" Cutting and boiling of 47 7
Raw materials, Chemical analysis of 46 27
" materials of coal gas 54 4
Receiving tanks for petroleum 52 8
Recovery of soda 47 30
Reducing of crude oils 52 41
Refined oil, Preparation of barrels for 51 32
" oil, Shipment of in tin cans 51 33
" oil, Transportation of 51 32
Refining of naphtha 52 27
" of sugar 50 64
" petroleum products 52 1
Relation of chemistry to cement manufacture 45 4
Relief holder 55 15
Rescission generator 55 26
Revivification of charcoal 50 68
Riehle's briquet-breaking machine 46 11
Rock-emery mill for pulverizing natural cements 45 23
Roll grease 52 54

Sec. Page
Rosin, Analysis of 48 37
" size, Analysis of 48 38
" size for paper 47 71
Rotary drying furnaces for soda recovery 47 33
" kilns 45 54

S Sec. Page
Sampling cement 46 38
" of sugar and molasses 50 70
Sand and cement briquets 46 10
Saturation or second carbonation of sugar-beet juice 50 31
Seasoning cement 46 37
Second carbonation or saturation of sugar-beet juice 50 31
" sugar 50 49
Selection of raw materials 46 41
Separation of sugar crystals from molasses by centrifugals 50 49
Setting, Effect of temperature and time of 46 6
" Indications of 46 4
" time of Portland cement 46 2
Settings, Primary and secondary 46 5
Schweitzer's reagent 47 3
Screens for washing wood pulp 47 21
Scrubbers 54 26
Shaft kilns 45 50
Shoefer kiln 45 52
Shredding and crushing sugar cane 49 12
Shutting down of retorts 54 16
Silica Portland cement, Manufacture of 45 31
" Portland cement, Properties of 45 31
Sirups 50 62
Sizing paper 47 71
Slaked lime 45 12
Slaking lime, Importance of 45 13
Slag cement 45 26
" cement, Composition of 45 28
" cement, Manufacture of 45 27
" Portland cement, Manufacture of 45 29
Slags, Adaptability for cements 45 26
" Composition of 45 27
Slicing the sugar beets 50 22
Slitting and rewinding machine for paper 47 89

	Sec.	*Page*
Sludge from causticizing pans, Analysis of	48	17
Slurry process	45	59
Soap stock, Congealing point of	53	25
Soda ash, Analysis of	48	11
" process for wood pulp	47	15
" pulp, Analysis of	48	52
" pulp mill, Ground plan of	47	27
" recovery, Calculation of	47	35
" recovery of	47	30
" recovery, Rotary drying furnaces	47	33
Sodium chloride, Analysis of	48	41
Sources of light for photometers	55	39
Specific gravity of cement	46	21
Specifications for tensile strength	46	16
Speed of breaking briquets	46	13
Spot and star diaphragms	55	38
Spray condensers	52	12
" generator	55	26
Standard solutions for paper analysis	48	5
State requirements for oil	53	35
Stevens process for sugar-beet molasses	50	57
Stills for refining petroleum	52	1
Storage apparatus for acetylene gas	55	28
" tanks for petroleum	52	8
Storing of sugar beets	50	9
Straw	47	4
" Bleaching of	47	56
" pulp	47	12
Strontia processes for sugar-beet molasses	50	58
Sugar-beet factories, Lime kilns in	50	35
" beet juice, Calorization of	50	29
" beet juice, Carbonation of	50	30
" beet juice, Concentration of	50	41
" beet juice, Crystallizers for	50	49
" beet juice, Difficulties in carbonation of	50	32
" beet juice, Evaporators for	50	43
" beet juice, Measurement of	50	28
" beet juice, second carbonation or saturation of	50	31
Sugar-beet juice, Sulphuring of	50	41
" beet juice, Vacuum pan for	50	45
" beet molasses, Composition of	50	52
" beet molasses, Elution process of	50	56
" beet molasses, Osmose process of	50	53
" beet molasses, Stevens process of	50	57
" beet molasses, Strontia processes of	50	58
" beet molasses, Triple Osmose process of	50	55
" beet plows	50	6
" beet sludge, Filter pressing of	50	38
" beets	50	2
" beets, Beds for	50	7
" beets, Chemical constituents of	50	10
" beets, Cultivation of	50	4
" beets, Diffusion of	50	23
" beets, Harvesting of	50	5
" beets, Planting of	50	4
" beets, Storing of	50	9
" beets, Slicing of	50	22
" beets, Washing of	50	18
" cane	49	4
" cane, Crushing of	49	9
" cane, Defecation of	49	20
" cane, Diffusion of	49	19
" cane juice, Clarification by Howard's process of	49	24
" cane juice, Concentration and granulation of	49	30
" cane juice, Defecation and clarification by lime of	49	21
" cane juice, Defecation by calcium bisulphide of	49	26
" cane juice, Defecation by sulphurous acid and lime of	49	27
" cane juice, Preliminary straining of	49	19
" cane, Maceration of	49	16
" cane, Shredding and crushing of	49	12
" Crystallizable and uncrystallizable	49	5

Sec. *Page*

Sugar crystals, Separation of molasses by centrifugals.... 50 49
" Decolorization of, by charcoal.... 50 66
" Definition of.... 49 1
" Dutch standards of.... 50 72
" Granulation of, in vacuum pan.... 50 45
" History of.... 49 2
" manufacture, Synopsis of.... 49 9
" refining.... 50 64
" Second.... 50 49
" Third.... 50 19
Sugars and molasses, Sampling of.... 50 70
Sulphate process of wood pulp 47 52
Sulphite digesters.... 47 18
" liquor, Disposition of waste of.... 47 49
" process, Bisulphite liquor.... 47 36
" process, Sulphur burners.... 47 37
" process of wood pulp 47 36
" pulp, Analysis of.... 48 53
" pulp, Bleaching of.... 47 58
" pulp mill, Ground plan of.... 47 49
Sulphur, Analysis of.... 48 43
" burners, Sulphite process.... 47 37
" dioxide, Absorption of.... 47 30
" dioxide, Manufacture of.... 47 37
" Qualitative test for, in oils.... 58 17
" Quantitative determination in petroleum of.... 58 17
Sulphuric-acid refining test of petroleum.... 58 19
Sulphuring sugar-beet juice... 50 41
Sulphurous acid, Generation of 49 29
Sunning of crude oils.... 52 41
Supercalendering of paper.... 47 86

T *Sec.* *Page*

Tagliabue closed-cup tester... 58 8
" open-cup tester.... 58 15
Tank cars.... 51 30
Tar extractor.... 54 22
" separator.... 55 11
Techno-chemical examination of crude petroleum.... 51 8
Tempering molasses.... 49 44
Tempers.... 54 11
Tensile strength, Comparisons of.... 46 16
" strength of various cements.... 46 15
" strength, Specifications for.... 46 16
" strength, Test of.... 46 7
Test of compressive strength.. 46 16
" of tensile strength.... 46 7
Testing briquets in series.... 46 10
" cement, Points to be observed in.... 46 14
" of paper.... 48 54
" of Portland cement.... 46 1
Tests on natural cements.... 45 24
Third sugar.... 50 49
Torpedoing oil wells.... 51 28
Transportation of oil.... 51 29
" of refined oil.. 51 32
Treatment of bleached stock.. 47 65
Tub sizing of paper.... 47 85
Types of acetylene-gas generators.... 55 25

U *Sec.* *Page*

Ultramarine, Analysis of.... 48 34
United States quartermaster's specifications for oils.... 58 31
Unsoundness of cement.... 46 18
Use of lubricants.... 52 52
Uses of cement.... 46 43

V *Sec.* *Page*

Vacuum pan for sugar-beet juice.... 50 45
" pans.... 49 34
" pans, Working of... 49 40
Vaseline.... 52 55
Vinasse or molasses residue... 50 63
Viscosimeter, Saybolt.... 58 22
" Tagliabue's improved.... 58 22

W *Sec.* *Page*

Warm-water test.... 46 18
Warren filter for water.... 47 95
Washer, Worm.... 47 47
Washers.... 54 25
Washing of wood pulp.... 47 19
" the sugar beets.... 50 18
Water, Alum treatment of.... 47 93

	Sec.	*Page*
Water. Filtration of	47	92
" gas apparatus	55	5
" gas apparatus, Drying of	55	6
" gas apparatus, Fire-brick lining of	55	14
" gas apparatus, Firing of	55	9
" gas apparatus, Shutting down of	55	10
" gas apparatus, Starting of	55	6
" gas apparatus, Working of	55	7
" gas, Composition of	55	2
" gas, Manufacture of	55	3
" gas, Oil for	55	18
" Jewell filter for	47	95
" Purification of	47	92
" Warren filter for	47	95
Waxes	52	56
Wet station meters	54	82
" station meters, Testing of	54	32

	Sec.	*Page*
White size for paper	47	72
Whitehall raw-stone mill	45	48
Wood	47	4
" Chipping of	47	15
" fibre, Bleaching of	47	57
" Grinding of	47	13
" Preparation of, for the sulphite process	47	43
" pulp	47	13
" pulp, Analysis of	48	51
" pulp by Barre and Bondel's nitric-acid process	47	52
" pulp by nitrohydrochloric-acid process	47	52
" pulp by Pictet and Brélaz's process	47	52
" pulp, by sulphate process	47	52
" pulp, Preparing of	47	13
" pulp, Soda process	47	15
" pulp. Sulphite process	47	36
Working of vacuum pans	49	40
Worm washer	47	47

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